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NBS SPECIAL PUBLICATION **333**

**Research Materials
Developed Under the
NBS Inorganic
Materials Program**

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NATIONAL BUREAU OF STANDARDS • LEWIS M. BRANSCOMB, *Director*

Research Materials Developed Under the NBS Inorganic Materials Program

Edited by
F. E. Brinckman and J. B. Wachtman, Jr.

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Research Materials Developed Under the NBS Inorganic Materials Program

F. E. Brinckman and J. B. Wachtman, Jr.

The National Bureau of Standards develops many specialized materials in the process of carrying out research supporting its measurements, standards, and service activities. These materials include gases, liquids, glasses, single crystals, polycrystals, and various multiphase materials; their compositions (including trace elements in some cases) and physical characteristics are tailored to specific research needs, but the materials are often of use for other research purposes. Production is usually limited to immediate internal needs and samples are not generally available for distribution, but knowledge of production techniques and materials characteristics which may be helpful to other scientists is available. The present listing of research materials developed in the Inorganic Materials Division accordingly gives names of scientific staff members who may be contacted for this type of information as well as giving a brief summary of the nature, method of preparation and properties determined for each material.

Key words: Chemical properties; composition; gases; glasses; liquids; multiphase materials; physical properties; polycrystals; preparation; research.

1. Introduction

Improved materials are critically needed in many areas of technology. Attempts to provide such superior materials are frequently empirical and sometimes wasteful of time and funds [1].¹ An empirical component will probably always be present in any materials development program, but the establishment of a science of materials that increasingly affords predictable and reliable results in devising new materials for specific tasks is most desirable. Not only will this increase the efficiency of materials development programs, but also can ultimately lead to development of completely new materials or new applications. Much thought has been given to opportunities arising from recent developments in materials science [2] and to roadblocks preventing engineering achievement of scientifically feasible materials including the areas of characterization [1] and processing [3]. Advances in materials science assist materials development on the one hand, but depend upon development of materials suitable for research on the other. This interdependence of materials development and materials science is widely recognized and new programs on the properties of materials increasingly have as a major component a research material development subprogram. The experimental and theoretical competence needed to produce and characterize specimens is frequently quite different from that needed for the subsequent property study so that the decision to undertake a property study on a new material frequently means that a large investment of time and resources, perhaps comparable to the property measuring effort itself, must be made before the property study can begin. In these circumstances the availability of a few trial specimens, even if not of completely satisfactory quality for the final study, can be very valuable in permitting a test of the experimental feasibility of the contemplated measurements and perhaps in providing guidance on how closely the character of the specimen must be controlled for the final study. When trial specimens are not available, knowledge of previous preparation techniques and of the nature and quality of specimens which have

been produced successfully is usually quite valuable in planning and carrying out the sample preparation aspect of a new research program. The present listing of research materials produced in the Inorganic Materials Division is offered as a means of promoting direct scientist-to-scientist exchange of information on production and characterization of materials for which the division has some special competence. In some cases, specimens may be on hand and available on request; in other cases, facilities and staff may be available to produce additional specimens for purposes coming within the NBS mission. Provision of information, rather than specimens, is the principal goal, however, because maintenance of a stock of the numerous and extremely varied materials produced in small batches or maintenance of specialized production facilities on a standby basis is not feasible.

Information concerning nationwide sources of many research materials, especially single crystals, is available from the Oak Ridge Research Materials Information Center [4].

The present listing of materials may be useful to scientists and engineers interested in the production of a material or its general availability for purposes other than research. No claim is made concerning exhaustive knowledge of the materials listed, but the staff members involved in the production or characterization of unusual materials sometimes have special knowledge relevant to other applications in addition to research.

2. Definition of Research Materials

2.1. General Definition

A research material is here considered to be one sufficiently well-characterized to be useful for a particular type of current research. Ideally, a fully characterized material is desirable; that is, the character (chemical composition, structure, microstructure, etc.) should be uniform and held within such narrow and known limits that all the chemical and physical properties of the material are well determined. Practical considerations usually limit the characterization to

¹ Figures in brackets indicate the literature references on page 2.

the factors thought to control the property being studied and limit the accuracy of the characterization. Thus, a material useful at a particular stage of research may no longer be useful for the same type of research as measurement techniques improve and finer details of behavior are studied or as new factors are discovered to have an influence on properties at the level being studied. A valid research material can cease to have this distinction as a field of study progresses but the same material can sometimes again become an important research material as new phenomena are investigated. For example, ruby grown by the Verneuil process for bearings, wear surfaces, or jewels suddenly became an exciting research material when the ruby laser was invented.

2.2. Differences in Degree of Characterization Needed for Research on Properties with Varying Degrees of Structure Sensitivity.

The characterization needed for certain research, such as phase equilibria, is sometimes limited primarily to the major element composition and such factors as trace impurities or surface condition are relatively unimportant. This fact is reflected in the listing, for example, of a number of crystalline materials developed for phase equilibria studies and a number of glasses developed for bulk optical or elasticity studies; major component characterization was sufficient for these purposes. Some of these materials have subsequently become the object of renewed research interest in connection with other properties dependent primarily upon major component composition such as electro-optic properties, photoelasticity, and mechanical properties under pressure. Certain structure-sensitive properties, such as transport, plastic deformation, and fracture depend upon trace impurities and upon small deviations from stoichiometry. Samples made for research in these areas, even though of the same major component composition as some of those mentioned previously, require more careful processing and characterization and are accordingly listed as separate entries with an indication of their special features.

2.3. Inhomogeneity

Among both the bulk composition and trace impurity types there are cases where a deliberate degree of inhomogeneity is required. Thus, a series of glasses with the same total composition but differing degrees of phase separation forms an interesting family of research materials. An example of an even finer scale of deliberately produced inhomogeneity is a series of crystals of CaF_2 all having the same level of Gd additive but differing in the degree of association of point defects with Gd atoms. This last example illustrates another feature of some of the listed materials; in this case specimens of given bulk composition were produced by a commercial supplier to NBS specifications. The NBS contribution was the development of special treatments and measurement tech-

niques needed to produce and determine different degrees of association.

2.4. Metastable Materials

Still another category of research materials involves those which are not stable or have a very short lifetime under ordinary conditions but which occur as important components under special conditions such as high temperature vapor species, high pressure crystalline phases, or short-lived reactive intermediates. Such materials generally cannot be kept in stock but the technology of preparation and the techniques for measurement of concentration and properties during the brief lifetime of the material are important aspects of research material information. An overlapping family of research materials consists of those which are dangerous (toxic, explosive, or corrosive) and so require special handling procedures during processing, storage, or property measurement. An example of such a difficult research material is the high pressure polymorph of the detonator explosive lead azide.

3. Relation to Standard Reference Materials

A few of the research materials listed here have been developed into Standard Reference Materials and many of the Standard Reference Materials are used as research materials but the two categories should not be confused despite the fact that there is some overlap. Standard Reference Materials are kept in stock by the National Bureau of Standards and sold by the Office of Standard Reference Materials. Each Standard Reference Material is certified with respect to the aspects of chemical and/or physical properties relevant to the material's intended application. Perhaps the greatest use of these materials is for calibration of instruments and checking of measurement procedures, but the high degree of homogeneity and extensive characterization associated with Standard Reference Materials frequently makes them useful as research materials. A full listing of Standard Reference Materials is available [5].

4. Organization of the Tables

4.1. Rationale

As noted in the Introduction, entries have been tabulated into sections derived chiefly from gross physical state at ordinary conditions (e.g., gases, polycrystals, multiphases, etc.). Clearly a number of exceptions occur, particularly for research materials of low stability or transient existence, but their location will be fairly obvious to the reader.

Since this compilation tends to emphasize composition rather than properties, special care was taken in organizing the tables. Use of the widely accepted Chemical Abstracts Formula Index does not always generate familiar empirical chemical formulae, nor

does this system readily lend itself to classification of glasses, but we chose it for its rational basis. Moreover, significant progress has been achieved for computer searching and retrieval; indeed, the parent program is now in use by the Patent Office.

4.2. Use of Tables—Key

a. The arrangement of symbols in formulae is alphabetic except that in carbon compounds C always comes first followed immediately by H if hydrogen is also present.

b. The arrangement of formulae or entries is also alphabetic, except that the number of atoms of any specific kind influences the order of listing; e.g., all C_1 compounds appear before C_2 , thus CCl_2O , CCl_4 , $CHCl_3$, CHN , CH_2O , CO , C_2Ca , $C_2H_4O_2$.

c. Water of hydration is not made a part of the formulae indexed.

d. Polymers having different names and recognized as different substances, e.g., acetaldehyde and paraldehyde, are all entered under their accepted formulae; but a definite compound for which different polymeric formulae are in use is entered under the simplest formula.

e. For series of compounds listed under a single entry heading "M" refers to metallic or metalloid components (e.g., K, Li, Rb) and "X" denotes electronegative substituents such as Cl, F, O, S, etc.

f. Approximate compositions are indicated by enclosing listing in quotation marks, "—", such as in interstitial compounds or alloys.

g. Glasses, polyphases, and compounds of given stoichiometry but indefinite structure are cited alphabetically by M, followed by X; thus $B_2O_3 \cdot 3Nb_2O_5$; $Cr_2O_3 \cdot IrO_2$; $B_2O_3 \cdot BaO \cdot SiO_2$; etc.

h. Isotopic compositions are presumed to be of terrestrial abundance unless otherwise indicated.

i. Wherever practicable conventional formulae and/or nomenclature is listed to the right of the index formula.

j. Great effort has been made to maintain accuracy and provide current information. In some instances, through staff changes, some authors cited are no

longer in the Inorganic Materials Division, yet inclusion of their recent efforts was deemed significant to this compendium. Hence, these individuals are signified by an asterisk. The reader may contact these individuals for further information, but it would probably be more expedient if the editors were contacted.

Many of the materials listed in this report were produced as part of programs sponsored by other agencies. Acknowledgements for specific materials are given in the references and are too numerous to repeat in detail here. The support by the Air Force Materials Laboratory, the Air Force Cambridge Research Laboratory, the Army Research Office (Durham), the Advanced Projects Research Agency (Materials Office), the Atomic Energy Commission (Research Division), the National Aeronautics and Space Administration, and the Office of Naval Research is gratefully acknowledged.

5. References

- [1] Characterization of Materials, prepared by the Committee on Characterization of Materials of the Materials Advisory Board, H. W. Leverenz, Committee Chairman, National Academy of Sciences—National Academy of Engineering Publication MAB-229-M, March 1967.
- [2] Research in Solid-State Sciences—Opportunities and Relevance to National Needs, prepared by a Committee of the Solid State Sciences Panel for the Coordinating Committee for Materials Research and Development of the Office of Science and Technology, R. Smoluchowski, Committee Chairman, Publication 1600, National Academy of Sciences, 1968.
- [3] Ceramic Processing, prepared by the Committee on Ceramic Processing, of the Materials Advisory Board, Joseph A. Pask, Committee Chairman, Publication 1576, National Academy of Sciences, 1968.
- [4] J. W. Cleland, Purity and Perfection of Research Specimens of Oxides in Mass Transport in Oxides, Nat. Bur. Stand. (U.S.), Spec. Pub. 296, (224 pages) edited by J. B. Wachtman, Jr. and A. D. Franklin, issued August 1968. The address of the Research Materials Information Center is Building 3001, Oak Ridge National Laboratory, Box X, Oak Ridge, Tennessee 37830.
- [5] Catalog of Standard Materials, Nat. Bur. Stand. (U.S.), Spec. Publ. 260, July 1970. See also supplements for price lists and changes.

I. GASES

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION		REFERENCES (Author, Title, Journal)
$\text{BCl}_n\text{F}_{3-n}$	equilibrium soln. of all species, where n = 0-3, studied as liquids	$\text{BCl}_3 + \text{BF}_3 \xrightleftharpoons[\text{at } < 25^\circ\text{C}]{}$	NMR (^{11}B , ^{19}F) spectrometry	attempt to determine chemical exchange rates in equilibrium	T. C. Farrar		(1) T. C. Farrar and T. D. Coyle, "Proton and Fluorine NMR Spectra of BF_2F_2 ", <i>J. Chem. Phys.</i> , <u>41</u> , 2612 (1964).
BF_2H	also studied as liquid	(3) BF_3^+ B_2H_6 at 250°C (1,2) (4) BF_3^+ $(\text{CH}_3\text{O})_2\text{BH}$ B_2F_4^+ $(\text{CH}_3)_3\text{SiH}$ + hv	mol. wt., infrared NMR (^1H , ^{11}B , ^{19}F) spectrometry, active H, chemical reactivity	qualitative structural dets; only known mono- boron fluoro- hydride; synth. reagent; air/ moisture sensitive	T. D. Coyle T. C. Farrar		(2) E. B. Whipple, T. H. Brown, T. C. Farrar, and T. D. Coyle, "Relative Signs of Nuclear Spin Coupling in $^{11}\text{B}^{19}\text{F}_2$ ", <i>J. Chem. Phys.</i> , <u>43</u> , 1841 (1964).
HBF_2	difluoroborane						(3) T. D. Coyle, J. J. Ritter, and T. C. Farar, "Prepar- ation and Properties of Difluoroborane", <i>Proc. Chem. Soc.</i> , <u>25</u> , (1964).
							(4) T. D. Coyle, J. Cooper, and J. J. Ritter, "Preparation and Some Reactions of Difluoroborane", <i>Inorg. Chem.</i> , <u>7</u> , 1014 (1968).

electric discharge rxn of B₃ in SiO₂ reactor infrared, mass spectrometry using full isotopic anal.

(1) F. E. Brinckman novel Si-²⁹E. E. Brinckman molecular structure bond angle and dissn. studies; unstable at r.t.

and G. Gordon, "Inorganic Intermediates in Inorganic Synthesis: Reactions of Covalent Main Group Fluorides in Electric Discharges", Fourth Int. Symp. on Fluorine Chem., Estes Park, Colo., July 1987, Abstr. of Papers, p. 132.

(2) F. E. Brinckman and G. Gordon, "Inorganic Intermediates in Inorganic Synthesis: Characterization of Transport Species in Electric Discharges", Proc. Int. Symp. on Decomposition of Organometallic Compounds to Refractory Ceramics, Metals, and Metal Alloys, Univ. Dayton Press (1987), p. 29.

B₂F₄, also studied as liquid diboron tetrafluoride tetrafluorodiborane-4

(1) F. E. Brinckman and G. Gordon, "Inorganic Intermediates in Inorganic Synthesis: Reactions of Covalent Main Group Fluorides in Electric Discharges", Fourth Int. Symp. on Fluorine Chem., Estes Park, Colo., July 1987, Abstr. of Papers, p. 132.

B₂F₄0 BF₂0BF₂
obsd. as parent-daughter ions

See refs. 1, 2 under BF₃OSi.

B₂Cl₄ + SbF₃ at -70°C infrared, mass NMR (¹¹B, ¹⁹F) spectrometry

simplest polyboron fluoride; potential source of BF₂ function in free radical reactions; B-B bond dissn. energy

T. D. Coyle T. C. Farrar V. H. Dibeler and S. K. Liston, "Mass Spectrometric Study of Photoionization. XII. Boron Trifluoride and Diboron Tetrafluoride", Inorg. Chem., 7, 1742 (1968).

novel B-O-B molecular structure suitable for bond angle and dissn. measurements; unstable even at low pressures

$n_{\text{B}_2\text{H}_6}$	studied as liquid diborane	$n_{\text{BF}_3} \cdot \text{O}(\text{C}_2\text{H}_5)_2 + \text{LiAlH}_4$ ($n = 10$ or 11)	infrared (1), NMR ($^1\text{H}, ^{10}\text{B}, ^{11}\text{B}$) spectrometry (2)	parent material for synthesis of selected isotope ($\text{H}, ^{10}\text{B}, ^{11}\text{B}$) boranes; detailed interpretation of NMR spectra	T. D. Coyle T. C. Farrar R. B. Johannessen	(1) W. J. Lafferty, A. G. Maki, and T. D. Coyle, "High Resolution Infrared Spectrum and Structure of Diborane", <i>J. Mol. Spectroscopy</i> , 33, 345 (1970).
CCl_3F	also studied as liquid, solid	commercial	infrared, NMR ($^{19}\text{F}, ^{35}\text{Cl}, ^{37}\text{Cl}$) spectrometry, volatility, m.p.	model compound for structure in liquid anisotropic motions in liquids; angular momentum cross-sections	T. C. Farrar	(2) T. C. Farrar, R. B. Johannessen, and T. D. Coyle, "Magnetic Non-Equivalence in the High Resolution NMR Spectra of Diborane", <i>J. Chem. Phys.</i> , 49, 281 (1968). See also $\text{C}_2\text{H}^{11}\text{BD}_6\text{O}_2$ in SECTION II.
CHN	Freon-11			used for photoionization studies: parent compound for X-CN series	T. D. Coyle	V. H. Dibeler and S. K. Liston, "Mass Spectrometric Study of Photoionization IX. Hydrogen Cyanide and Acetonitrile", <i>J. Chem. Phys.</i> , 48, 4765 (1968).
CH_3NO_2	methyl nitrite	$\text{NaNO}_2 + \text{CH}_3\text{OH} + \text{H}_2\text{SO}_4$	infrared, ultraviolet, mass spectrometry, vapor phase chromatography	prepd. for photoionization study	T. D. Coyle	

C_2HBCl_2
 $\text{trans}-\text{ClCH}=\text{CHBCl}_2 + \text{H}_2$
 ethynylchloro-
 borane

C_2HBF_2
 C_{2v} symmetry
 $(\text{CH}_3)_3\text{SnC}\equiv\text{CH} + \text{BF}_3$ at
 -80°C
 ethynyldifluoro-
 borane

$\text{ClFO}_3\text{FClO}_3$
 perchloroyl
 fluoride

$\text{C}_2\text{HCl}_2 + \text{H}_2$
 ethynylchloro-
 borane
 $\rightarrow \text{C}_2\text{H}_2 + \text{HCl}$.
 suitable for
 studies on
 coordination
 saturation of
 B; air/moisture
 sensitive

mass, infrared
 spectrometry;
 cleavage with
 propionic acid
 $\rightarrow \text{C}_2\text{H}_2 + \text{HCl}$.
 first ethynylboron
 chloride isolated;
 novel substituted
 acetylene;
 suitable for
 studies on
 coordination
 saturation of
 B; air/moisture
 sensitive

mass, infrared,
 and microwave
 spectrometry, cleavage
 in glass with
 propionic acid +
 $\text{C}_2\text{H}_2 + \text{SiF}_4$
 first ethynylboron
 fluoride isolated;
 suitable for extensive
 structural
 analysis; air/
 moisture sensitive

mass, infrared-
 nyboron iso-
 lated; suitable
 for extensive
 structural
 analysis; air/
 moisture sensitive

J. J. Ritter
 T. D. Coyle

J. J. Ritter
 T. D. Coyle

see CCl_3F

T. C. Farrar

J. J. Ritter
 T. D. Coyle

W. J. Lafferty, J.
 J. Ritter, "Microwave
 Spectrum, Structure,
 and Dipole Moment
 of Ethynylidifluoro-
 borane, $\text{HC}\equiv\text{CF}_2$ ",
 Chem. Comm., 909

(1969).

(1) R. B. Johannessen,
 F. E. Brinckman,
 F. E. Brinckman, and
 T. D. Coyle, "Synthesis
 of Ethynylboron
 Halides", Chem.
 Comm., 908 (1969).

(2) See refs. under
 BF_5Si_1 .

(3) See ref. under
 F_4Si_1 .

D $\text{D}(\text{atoms})$

microwave discharge in $\text{D}_2\text{O}(\text{g})$

measurement of electron g-factor relative to g_J of H atoms

$\text{PF}_2\text{I} + \text{HI}$ (1) mol wt; infrared NMR (^1H , ^{19}F , ^{31}P) (2)

specrometry

HF_2P
difluorophosphine

M. Linzer

experimental test of quantum electrodynamic calculations

M. Linzer, "Measurement of the Proton g-factor in Cyclohexane and the Electron g-factors in Atomic Deutrium and Atomic Nitrogen", Bull. Am. Phys. Soc., 12, 507 (1967).

5

F_3OP OPF_3 studied as liquids also commercial; fractional distillation infrared, mass (1), NMR (^{19}F , ^{31}P) (2) volatility; reagent uses; air/moisture sensitive

F_3P PF_3

Also obsd. as liquid (1)

SiF_4
tetrafluorosilane

T. D. Coyle
NMR relative signs; reagent uses; air/moisture sensitive

F. E. Brinckman, T. C. Farrar (2) study of electric discharge rxns in PF_3 (1); model compounds for study of structures and electron anisotropies (2)

T. D. Coyle, R. B. Brinckman, and T. C. Farrar, "Formation of Oxyfluorophosphines and Polyphosphines in Gaseous Discharge Reactions", 4th Middle Atlantic Regional Mtg of the Am. Chem. Soc., Wash. D. C., February 1969, Abstr. of Papers, p. 41.

T. D. Coyle, R. B. Brinckman, P. E. Brinckman, and T. C. Farrar, "NMR Studies of Inorganic Fluorides. IV. Relative Signs of Coupling Constants in CH_3SiF_3 , HSiF_3 , and HF_2SiF ", J. Chem. Phys., 47, 3088 (1967).



obsd as parent and daughter ions



microwave discharge rxns. of GeF_4 and SiF_4 in SiO_2 reactors

F_6OSi_2

obsd as direct inlet mass specrometry, full isotopic anal.

F_6OSSi_2

also studied as liquid

$Si_2Cl_6 + SbF_3(1)$; electric discharge rxn of SiF_4 with $SiO_2(2)$

Si_2OF_6 hexafluoro-disiloxane

F_6GeSiF_3

microwave discharge rxns. of GeF_4 and SiF_4 in SiO_2 reactors

NMR [$^{19}F, (^{29}Si)$] (1); mass (2) spectrometry suitable for vapor phase chromatography; air/moisture sensitive

$Si_2OCl_6 + SbF_3(1)$; mass (2) spectrometry of $Si-O-Si$ bond angle; vapor phase chromatography; air/moisture sensitive

(2) See ref. 2 under BF_3OSi_2 .

F_6Si_2

also studied as liquid

$Si_2Cl_6 + SbF_3(1)$

NMR [$^{19}F, (^{29}Si)$] (1); spectrometry (2) chemical reactivity (2) parent compound for perfluorodisilanyl chemistry; suitable for detm. of Si-Si bond energy; purified by vapor phase chromatography; air/moisture sensitive

Si_2F_6 hexafluorodisilane

(1) See ref. 1 under F_6OSi_2 .

(2) F. E. Brinckman, T. D. Coyle, and L. Fishman, "Formation of a Perfluorodisilanyl-silazane: Selected Cleavage Reactions of Silazanes", Fourth Int. Conf. on Organometallic Chemistry, Bristol, England, July 1969, Abstr. of Papers, p. D14.

Ga_2O

Ga_2O_4W

Ga_2Mo_4

N N(atoms)

$Ga_2O_3(s)$ heated to 1200-1500°C

$Ga_2O_3(s) + W$ heated to 1200°C

microwave discharge in $N_2(g)$

mass spectrometry

mass spectrometry

see D

see D

R. C. Paule

R. C. Paule

M. Linzer

See ref. under D.

III. LIQUIDS

COMPOSITION	NATURE OF MATERIAL	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION			REFERENCES (Author, Title, Journal)
				NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author, Title, Journal)		
$\text{BD}_{3-n}\text{F}_3\text{H}_n\text{P}$ $\text{F}_3\text{P}\cdot\text{BH}_n\text{D}_{3-n}$	$n = 1-3$ (2)	$\text{PF}_3+\text{B}_2\text{H}_6$ (deuterated)	synthesis, microwave spectrometry	rotational spectrum, barrier to internal rotation	T. D. Coyle	R. Kuczkowski and D. Lide, "Microwave Spectrum, Structure, Dipole Moment, and Barrier to Internal Rotation of Phosphorus Trifluoride-Borane", J. Chem. Phys., <u>46</u> , 357 (1967).	
$\text{BF}_7\text{Si}_2^2\text{SiF}_3\text{SiF}_2\text{BF}_2$	prep'd. at Rice Univ. (a) SiF_2 (g) + BF_3 (g) (b) co-condensed at -196°C	infrared, NMR ($^{11}\text{B}, ^{19}\text{F}$) spectrometry	series of novel Si-B compounds; qualitative determination of structure; air/moisture sensitive	F. E. Brinckman T. D. Coyle T. C. Farrar	P. L. Timms, T. C. Ehlert, J. A. Margrave, F. E. Brinckman, T. C. Farrar, and T. D. Coyle, "Silicon-Fluorine Chemistry. II. Silicon-Borane Fluorides, J. Am. Chem. Soc., <u>87</u> , 3819 (1965).		

B_2Cl_4	unstable liquid	infrared, mass spectrometry; chemical reactivity (3)	J. J. Ritter parent material for chemistry of diboron compounds; detm. of B-B bond energy	(1) T. Wartik, et al., "Diboron Tetrachloride", Inorg. Syn., <u>10</u> , 118 (1967); (2) V. H. Dibeler and J. A. Walker, "Mass Spectrometric Study of Photoionization. XIII. Boron Trichloride and Diboron Tetrachloride", Inorg. Chem., <u>8</u> , 50 (1969); (3) T. D. Coyle and J. J. Ritter, "Reactions of Diboron Tetrahalides with Some Unsaturated Organometallic Compounds", J. Organomet. Chem., <u>12</u> , 269 (1968).
$\text{Br}_n\text{F}_{3-n}\text{Si}$	$n = 1-3$ studied as liquids and gases	$\text{SiF}_4 + \text{SiBr}_4 + \text{AlBr}_3$ catalyst at 150°C	infrared, mass spectrometry; NMR [^{11}F (^{29}Si)] (1,2) spectrometry; volatility	F. E. Brinckman T. D. Coyle dipole moments for SiF_3Br and SiBr_3F (with A. Marryot, NBS)
				(1) T. D. Coyle, P. B. Johannessen, F. E. Brinckman, and T. C. Farrar, "Nuclear Magnetic Resonance Studies of Inorganic Fluorides. II. Solvent Effects on $J(^{29}\text{Si}-^{19}\text{F})$ in Silicon Terra-fluoride", J. Phys. Chem., <u>70</u> , 1682 (1966); (2) R. B. Johannessen, F. E. Brinckman, and T. D. Coyle, "Nuclear Magnetic Resonance Studies of Inorganic Fluorides. V. Fluorosilanes", J. Phys. Chem., <u>72</u> , 660 (1968).

$\text{CH}_3\text{B}_2\text{Cl}_3$	unstable	$\text{B}_2\text{Cl}_4 + (\text{CH}_3)_4\text{M}$ (M = Ge, Sn or Pb)	infrared, mass spectrometry; cleavage rxns. with $\text{Ag}_2\text{O} + \text{NH}_3(\text{aq}) + \text{CH}_4 + \text{H}_2$	J. J. Ritter T. D. Coyle	T. D. Coyle and J. J. Ritter, "Recent Studies in Organoboron Chemistry. Some Chemical Consequences of the Boron Subhalides", Fourth Int. Conf. on Organo-metallic Chemistry, Bristol, England, July 1969, Proc. of the Conf., p. R1.
$^{13}\text{CH}_3\text{I}$	also studied as gas	commercial, high-vacuum degassed	infrared, NMR ($^1\text{H}, ^{13}\text{C}$) spectrometry; m. p.	T. C. Farrar	See also $\text{C}_2\text{H}_4\text{O}_2$.
$^{13}\text{CH}_4\text{O}$ [^{13}C]-methanol		n = 10,11; also studied as solid	$\text{CD}_3\text{OH} + \text{nB}_2\text{H}_6$	T. D. Coyle T. C. Farrar	(1) T. C. Farrar, J. Cooper, and T. D. Coyle, "Proton Broad-Line NMR Study of [$^{12}\text{H}_6$ Dimethoxy-[^{11}B]borane", Chem. Comm., 610 (1966). (2) N. Boden, H. S. Gutowsky, J. R. Hansen, and T. C. Farrar, "Nuclear Magnetic Relaxation Studies of $(\text{CD}_3\text{O})_2\text{BH}^{11}\text{F}$ ", J. Chem. Phys., 46, 2849 (1967). (3) T. C. Farrar and T. Tsang, "A Nuclear Magnetic Resonance and Relaxation Study of Dimethoxyborane", J. Res. NBS, 73A, 195 (1969).

$\text{C}_2\text{H}^n\text{BD}_6\text{O}_2$
 $(\text{CD}_3\text{O})_2\text{nBH}$



T. D. Coyle and J. J. Ritter, "Structure, Isomerization, and Cleavage of 1,2-Bis-(dichloroboryl)-ethylene", *J. Am. Chem. Soc.*, **89**, 5739 (1967).

infrared, mass, NMR spectrometry; cleavage rxn of dehalo-compound with $\text{Ag}_2\text{O} + \text{NH}_3\text{(aq)}$ \rightarrow trans-di(deutero)ethylene

established the mode of addition of B_2Cl_4 to C_2H_2 ; pointed up the utility of ^1H NMR while irradiating at the boron frequency in distinguishing between $1,1$ and $1,2$ isomers while demonstrating the dangers of assigning structures on the basis of J_{HH} ; demonstrated utility of $\text{Ag}_2\text{O}/\text{NH}_3\text{(aq)}$ as B-C cleaving agent with retention of configuration; air/moisture sensitive



elemental anal.
 $\text{C}_2\text{H}_2\text{Cl}_4 +$
 $2\text{B}_2\text{Cl}_4$ or
 $\text{HC}_2\text{H}_2\text{B}_2\text{Cl}_4$

J. J. Ritter, T. D. Coyle, "Formation of B_2Cl_4 and halo-olefins during the reaction between B_2Cl_4 and halo-olefins to produce a known compound; air/moisture sensitive"

(1) J. J. Ritter, T. D. Coyle, J. M. Bellama, "The Interaction of B_2Cl_4 with Halo-olefins", Abstracts, 156th National Meeting of Amer. Chem. Soc., Atlantic City, N. J., (1968) P-INOR 166;

(2) C. Chambers, A. K. Holliday, and S. M. Walker, " $1,1,2$ Tetra-kisdichloroboryl-ethane", Proc. Chem. Soc., 286 (1964).

$C_2H_5B_2Cl_2MX_3$ (N=C, Si, Ge, Sn; X=Cl, CH ₃)	involatile liq. or oils	$B_2Cl_4 + CH_2 = CHMX_3$	elemental anal.; mol. wt., rxn. ratios	J. J. Ritter T. D. Coyle provides series of model com- pounds for structure- reactivity relationships in vicinal- and geminal- substituted organo- metal systems	See ref. (3) under B_2Cl_4 .
$C_2H_5B_3Cl_6$		$CH_2 = CHCl +$ 2 B_2Cl_4 or $CH_2 = CHBCl_2$ + B_2Cl_4	complete elemental anal.; mol. wt.; infrared, NMR spectrometry, rxn. ratio	demonstrated a reaction pathway be- tween B_2Cl_4 and halo olefins contrary to earlier reports of no reaction; suggested "BCl _n " as a possible "reactive inter- mediate" respon- sible for the observed product; air/moisture sensitive	(1) See ref. under $C_2H_5B_2Cl_2MX_3$. (2) See ref. (1) under $C_2H_5B_4Cl_4$.
$C_2H_4O_2$		$CH_2 = CHCl +$ 1,1,2-tris(dichloro- boryl)-ethane	infrared, NMR (¹ H, ¹³ C) spectro- metry; m.p.	model com- pound for study of ¹³ C T_1 and T_2 values	See also ¹³ CH ₃ I and ¹³ CH ₄ O.

^a[¹³C]-acetic acid

$\text{C}_4\text{H}_10\text{F}_3\text{NSi}_2$	$[(\text{CH}_3)_3\text{Si}]_2\text{NH}$ + SiF_4	infrared, mass, NMR ($^1\text{H}, ^1\text{F}$) spectrometry, elemental anal., sub- sequent rxns.	studied as solid; liquid unstable at -45°C	see ref. (1)	NMR (^1H) spectrometry (1,2)	model com- pound for preparative route to perfluorosilazanes	F. E. Brinckman, T. D. Coyle, and L. Fishman, "Formation of a Perfluoro- disilanylsilazane: Selected Cleavage Reactions of Sila- zanes", Fourth Int. Conf. on Organo- metallic Chemistry, Bristol, England, July 1969, Proc. of the Conf., p. D14.
$(\text{CH}_3)_3\text{SiNHSiF}_3$	$[(\text{CH}_3)_3\text{Si}]_2\text{NH}$ + Si_2F_6						
$\text{C}_3\text{H}_{10}\text{F}_5\text{NSi}_3$	$[(\text{CH}_3)_3\text{Si}]_2\text{NH}$ + Si_2F_5						
$(\text{CH}_3)_3\text{SiNHSi}_2\text{F}_5$							
C_4HCO_4	$\text{HCO}(\text{CO})_4$						
$\text{C}_6\text{H}_2\text{FeO}_4$	$\text{H}_2\text{Fe}(\text{CO})_4$	studied as liquid			infrared, NMR (^1H) spectro- metry	candidate compound for Fe-H distance detm., J _{Fe-H}	F. E. Brinckman T. C. Farrar See C_5fMnO_5 .

$C_4H_9MnO_5$	supplied by Prof. R. N. Grimes, Univ. of Virginia, Charlottesville, Va.	NMR ($^1H, ^{11}B$) spectrometry	clarification of molecular structure	T. C. Farrar	R. N. Grimes, "A New Isomer of $C_2B_1H_5(C_3\text{-Dimethyl-1,2-dicarbacyclo-5-pentaborane(5)}$ ", J. Am. Chem. Soc., <u>88</u> , 1070 (1966).
C_5HMnO_5 $HMn(CO)_5$	also studied as solid	prepared at Mass. Inst. Tech., and at NBS by F. E. Brinckman	infrared, NMR (1H) spectroscopy	model compound for application of NMR in detm. metal-H bond distance; see also C_4HCoO_4	T. C. Farrar
C_5HReO_5 $HRe(CO)_5$					T. C. Farrar
$C_5H_15N_3Si$	$n = 3$	(a) $CH_3N_3 + CH_3MgCl$ (b) $a + (CH_3)_nSiCl_4-n$	infrared, NMR (1H) spectroscopy (^{12}C), elemental anal., mol. wt. at -40 to -80°C; see ref. (1)	parent compounds for synthesis of 1,3-dimethyl-1-triazeno-metal(loid) deriv.; novel bidentate ligand for coordination studies; air/moisture sensitive	F. E. Brinckman
$C_6H_16N_6Si$	$n = 2$	$(CH_3)_nSi(CH_3)_n-$ $N = [CH_3]_{4-n}$			(1) F. E. Brinckman, H. S. Haiss, and R. A. Robb, "Metal-Nitrogen Bonding, Covalent Complexes of 1,3-Dimethyltriazenes with Elements of Groups I, II, III, IV, and V", Inorg. Chem., <u>4</u> , 936 (1965). (2) F. E. Brinckman, K. Nelson, R. Barefoot, and H. Haiss, "Silicon-Nitrogen Bonding-Penta- and Hexacoordinated Intermediates in Methylsilazanes", in preparation.

$\delta_{\text{C}_6\text{H}_5}$, 116
benzene

$\delta_{\text{C}_6\text{H}_5\text{Cl}}$,
160, 35
pyridine

$\delta_{\text{C}_6\text{H}_5\text{Cl}}$,
160, 35
pyridine

NMR ($^1\text{H}, ^1\text{C}$)
spectrometry
of Fast-
Fourier trans-
form NMR
spectrometry to
natural abundance
 ^{13}C

application(s)
of Fast-
Fourier trans-
form NMR
spectrometry to
natural abundance
 ^{13}C

$\text{C}_6\text{H}_5\text{Cl}$
cyclohexane

M. Linzer,
"Measurement
of ^1H g-
factor rela-
tive to g_J of
H atoms;
secondary
standard for
g-factor detms;
check on cal-
culation of para-
shielding para-
meter for H_2
molecule

Cl_4V
 VCI_4

commercial,
spectro-
grade

NMR (^1H)
spectrometry

M. Linzer
measurement
of ^1H g-
factor rela-
tive to g_J of
H atoms;
secondary
standard for
g-factor detms;
check on cal-
culation of para-
shielding para-
meter for H_2
molecule

Cl_4V
 VCI_4

obsd, as
commercial,
fractionally
distilled
and in TiCl_4
soln.; also
obsd, as poly-
crystalline
solid

EPR spectrometry
(as a function of
frequency, temp.);
magnetic suscepti-
bility between
78-30°K; optical
spectrophotometry
of VCl_4 in TiCl_4

Cl_5FSi_2
 $\text{Si}_2\text{Cl}_5\text{F}$
pentachlorofluoro-
disilane

infrared, mass
NMR [^{19}F , (^{19}F - ^{29}Si)]
spectrometry
at 150°C

F. E. Brinckman
T. D. Coyle
first ex-
ample of
mixed per-
halogenated
disilane; ^{19}F
"tag" permits
double-
resonance NMR
of Si nuclei

$\text{Br}_n\text{F}_{3-n}\text{Si}_i$.
R. B. Johannesen, F.
E. Brinckman, and T.
D. Coyle, "Nuclear
Magnetic Resonance
Studies of Some
Fluorinated Mono-
and Polysilanes",
153rd Natl. Mtg.
Am. Chem. Soc.,
Miami Beach, Fla.,
April 1967, Abstr.
of Papers, p. L113.

See also $\text{C}_6\text{H}_5\text{R}_2$
Under SELECTION IV.

$\text{Cl}_{16}\text{Si}_2$
hexachloro-
disilane
"polywater"

commercial,
fractional
distillation;
electric dis-
charge in
 SiCl_4
 $\text{H}_2 + \text{Cl}^-$
rxn with $\text{H}_2\text{O} \rightarrow$

infrared, mass
(full isotopic
anal.), spectro-
metry; vapor
phase chromatog-
raphy; volati-
lity, cleavage
of Si-Si bond
energy detm.
air/moisture
sensitive

F. E. Brinckman, J.

Cooper, and T. D.
Coyle, "Interactions
of Some Halosilanes
with Nitrogen Bases",
153rd Natl. Mtg. Am.
Chem. Soc., Miami
Beach, Fla., April
1967, Abstr. of
Papers, p. L112.

See SECTION I under
 FeSi_{12} .

H_2O
polymeric
not available

infrared, NMR
(^1H) spectrometry

parent mat-
erial for
synthesis of
compounds
bearing Si-Si
bond; suitable
for Si-Si bond
energy detm.
attempt to
establish
nature of
protons in
sample (work
with M.
Maimberg, NBS)

F. E. Brinckman
 FeSi_{12}

T. C. Farrar
E. D. Lippincott

III. CLASSES

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBER ²	
					TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author, Title, Journal)
barium aluminoborofluorosilicate glasses; example: Al ₂ O ₃ , 12 mol% B ₂ O ₃ , 16 BaF ₂ , 20 BaO, 8 SiO ₂ , 44	microspheres, 5-50 μ dia.	melting, chilling, reduction; flame spherizing	refractive index, thermal expansion, sphere size	filler material for dental composites	G. W. Cleek	R. L. Bowen and G. W. Cleek, "X-Ray Opaque Reinforcing Fillers for Composite Material," J. Dental Res., 48, 79 (1969).
glasses from Al ₂ O ₃ -BaO-SiO ₂ system	glass blocks	melting, annealing	refractive index, transmittance, liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek	ASTM draft in preparation.
aluminosilicate glass, alkali-free	NBS Standard Reference Material # 715	melting, chilling	Knoop hardness	not NBS certified for this method but suitable as standard; used in ASTM round robin tests	A. Napolitano	
glasses from B ₂ O ₃ -BaO-SiO ₂ system	glass blocks	melting, annealing	chemical analysis; refractive index; density; liquidus temp.	specimens may be used for detm. of other properties	G. W. Cleek	E. H. Hamilton, G. W. Cleek, and O. H. Grauer, "Some Properties of Glasses in the System Barium Oxide-Boric Oxide-Silica," Amer. Ceram. Soc., 41, 209 (1958).
glasses from B ₂ O ₃ -Ln ₂ O ₃ system	small fragments	gravity separated from two immiscible liquids	polarizing microscopy	high-index solid state	E. M. Levin	E. M. Levin, "Liquid Immiscibility in the Rare Earth Oxide-Boric Oxide Systems," Phys. & Chem. Glasses, 7, 90 (1966).

glasses from $\text{B}_2\text{O}_3\text{Na}_2\text{O-SiO}_2$ system	glass blocks	melting, chilling	domain develop- ment kinetics; critical immiscibility temp.	W. Haller specimens used for theoretical studies of immiscibility	W. Haller tested as possible method to determine neutron flux	W. Haller "Glass Beads for Neutron Flux Measure- ments", NBS Technical Highlights, 1965, p. 86
^{10}B -enriched borate glasses doped with Co, Dy, or In	glass prisms	melting, chilling, grinding	neutron acti- vation anal.	D. H. Blackburn specimens may be used for dem. of other properties	D. H. Blackburn specimens are being evaluated for fluorescence lifetime	E. J. Sharp, M. J. Weber, and G. W. Cleek, "Energy Trans- fer and Fluorescence Quenching in Eu and Nd Doped Silicate Glasses", <i>J. Appl. Phys.</i> , <u>41</u> , 364 (1970).
glasses from $\text{BaO-Na}_2\text{O-SiO}_2$ system	glass blocks	melting, annealing	refractive index, trans- mittance, liquidus temp.	G. W. Cleek specimens may be used for dem. of other properties	G. W. Cleek specimens may be used for dem. of other properties	E. J. Sharp, M. J. Weber, and G. W. Cleek, "Energy Trans- fer and Fluorescence Quenching in Eu and Nd Doped Silicate Glasses", <i>J. Appl. Phys.</i> , <u>41</u> , 364 (1970).
a base glass: BaO 5 mol% Na_2O 15 SiO_2 75 ZnO 5 doped with various rare earths: Eu, Er, Nd	blocks $3 \times 3 \times 3$ cm.	melting, chilling, annealing	evaluation for laser use	G. W. Cleek specimens are being evaluated for fluorescence lifetime	G. W. Cleek specimens may be used for dem. of other properties	E. J. Sharp, M. J. Weber, and G. W. Cleek, "Energy Trans- fer and Fluorescence Quenching in Eu and Nd Doped Silicate Glasses", <i>J. Appl. Phys.</i> , <u>41</u> , 364 (1970).
glasses from $\text{BaO-Nb}_2\text{O-SiO}_2$ system	glass blocks	melting, annealing	refractive index, trans- mittance, liquidus temp.	G. W. Cleek specimens may be used for dem. of other properties	G. W. Cleek specimens may be used for dem. of other properties	E. J. Sharp, M. J. Weber, and G. W. Cleek, "Energy Trans- fer and Fluorescence Quenching in Eu and Nd Doped Silicate Glasses", <i>J. Appl. Phys.</i> , <u>41</u> , 364 (1970).
glasses from BaO-TiO_2 system	glass blocks	melting, annealing	refractive index, transmittance, liquidus temp.	G. W. Cleek specimens may be used for dem. of other properties	G. W. Cleek specimens may be used for dem. of other properties	E. J. Sharp, M. J. Weber, and G. W. Cleek, "Energy Trans- fer and Fluorescence Quenching in Eu and Nd Doped Silicate Glasses", <i>J. Appl. Phys.</i> , <u>41</u> , 364 (1970).

glasses from BaO-SiO ₂ -ZnO system	glass blocks	melting, annealing	refractive index, transmittance, liquids, temp.	specimens may be used for detm. of other properties	G. W. Cleek
alkali-lime- silica glass: CaO 11.6 mol% K ₂ O 7.7 Na ₂ O 8.7 SiO ₂ 70.5	NBS Standard Reference Material # 710	melting, chilling	viscosity by beam bending	not NBS certified for this method but suitable as standard	A. Napolitano (1) ASTM C598 and H. E. Hagy, "Calibration of Beam Bending Ap- paratus", J. Amer. Ceram. Soc., <u>46</u> , 93 (1963).
			surface tension at elevated temp.	not NBS certified for this method but suitable as standard	A. Napolitano (2) D. F. Secrist, "Measurement of Sur- face Tension of Glasses by a Capillary Flow Technique", Bull. Amer. Ceram. Soc., <u>48</u> , 563 (1963).
			Knoop hardness	not NBS certified for this method but suitable as standard; used in ASTM round robin tests	A. Napolitano (3) ASTM draft in preparation.
				viscosity by penetrometer	A. Napolitano (4) J. E. Kelley, T. D. Robert, and H. M. Hanes, "A Penetro- meter for Measuring the Absolute Viscosity of Glass", U. S. Bureau of Mines RI 6358 (1964).
Fe-containing silicate glasses	30 x 30 x 2 mm	melting, chilling		Mossbauer spectrometry	G. W. Cleek specimens covering a wide range of Fe content may be used for characterization by Mossbauer spectroscopy

alkali-lead-silica glass:
 K_2O 5.6 mol%
 Na_2O 2.5
 PbO 45.3
 SiO_2 46.0

NBS Standard Material # 711

surface tension at elevated temp.

not NBS certified for this method but suitable as standard

Knoop hardness

A. Napolitano ASTM draft in preparation.

glasses from $Na_2O-SiO_2-TiO_2$ system

melting, annealing

refractive index, transmittance, liquidus temp.

not NBS certified for this method but suitable as standard; used in ASTM round robin tests

porous silica glass
 SiO_2

melting, chilling, thermal domain development, differential dissolution

mercury intrusion porosimetry, nitrogen absorption

not NBS certified for this method but suitable as standard; used in ASTM round robin tests

G. W. Cleek specimens may be used for detm. of other glass properties

W. Haller possible surface area and porosity standards

E. H. Hamilton and G. W. Cleek, "Properties of Sodium Titanium Silicate Glasses", J. Res. NBS, 61, 89 (1958).

(1) W. Haller, "Rearrangement Kinetics of the Liquid-Liquid Immiscible Microphases in Alkali-Boro-silicate Melts", J. Chem. Phys., 42, 686, (1965).
(2) N. M. Winslow and J. J. Shapiro, "An Instrument for the Measurement of Pore-Size Distribution by Mercury Penetration", ASTM Bull, 39 (Feb. 1959).

G. W. Cleek prisms covering range of refractive indices 1.5 to 1.9 measured by minimum deviation method for N_C , N_D , and N_F

melting, chilling, annealing, polishing

refractive index

A. Napolitano ASTM draft in preparation.

NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	REFERENCES (Author, Title, Journal)
Al ₂ O ₃ alumina	also studied as powder and crystals, see SECTION V	(1) commercial powder and crystals (2) purified powder by solar furnace	melting point melting point detm. for SRM	S. J. Schneider and C. L. McDaniel, "Effect of Environment upon the Melting Point of Al ₂ O ₃ ", J. Res. NBS, <u>71A</u> , 317 (1967).
single crystal bar	commercial	compressibility to 10Kb	see also under BH ₃ O ₃ , SECTION V	C. E. Weir, "Compressibility of Eleven Inorganic Materials", J. Res. NBS, <u>69A</u> , 29 (1965).
3 x 8 x 20 mm	chemical vapor deposition, AlCl ₃ + [O] + Al ₂ O ₃ at 1700-1750°C	spectrochemical anal, optical microscopy, x-ray diffractometry	material of high physical perfection and chemical purity; suitable for diffusion studies, physical properties	H. S. Parker A. Harding, "Vapor Growth of Aluminum Oxide Bicrystals", Abstracts, ACCS-NBS Conference on Crystal Growth, Aug. 11-13, 1969, Washington, D. C., p. 42.
~ 1cm	flame fusion, prep'd. by D. E. Roberts	optical, EPR spectrometry	laser properties (optical and x-ray damage)	R. F. Blunt T. Chang C. M. Stickey, et al, "Color Centers and Ruby-Laser Output-Energy Degradation", J. Appl. Phys., <u>40</u> , 1792 (1969).
Pure and with following dopants: Cr, Mg+Cr, Ti+Cr, Ti, V, Mn+Cr, Mn, Mn+Cr, +Or, Fe+Cr				

$\text{Al}_{1-n}\text{Cr}_n\text{X}_8\text{S}_2 \cdot 12\text{H}_2\text{O}$ $n = 1$
 $\text{KAl}_{1-n}\text{Cr}_n(\text{SO}_4)_2 \cdot$
 $12\text{H}_2\text{O}$

grown from
aq. soln.

G. A. Candela,
see also
 $\text{CrH}_2\text{NO}_8\text{S}_2 \cdot$
 $12\text{H}_2\text{O}$

AsGa
GaAs
gallium
arsenide
highly
doped and
compensated
vapor phase
epitaxy on
GaAs substrate,
 $\text{GaCl}_3 +$
 $\text{AsCl}_3 +$;
prepared by
Monsanto Co.

spectral examination
of temp. dependence
of radiative
recombination
induced by
electrical
injection
across p-n
junction or by
optical
excitation

(1) N. N. Winogradoff
and H. Kessler, "White
Emission and Electrical
Characteristics of
Epitaxial GaAs Lasers
and Tunnel Diodes",
Solid State Comm.,
2, 119 (1964).

(2) N. N. Winogradoff,
K. Owen and R. Cuttnutt,
"The Radiative Band
Pinch Effect and
Temperature Dependence
of Radiative Recom-
bination in GaAs",
Int. J. Electronics,
22, 229 (1957).

(3) N. N. Winogradoff,
"Field Control of the
Quantum Efficiency of
Radiative Recombi-
nation in Semicon-
ductors", Phys. Rev.,
138A, 1562 (1965).

(4) N. N. Winogradoff
and A. H. Neill, "Band
Tailoring Effects and
the Temperature De-
pendence of Radiative
Recombination in
Compensated Epitaxial
GaAs Laser Junctions",
IEEE J. Quant.
Electronics, 4,
111 (1968).

$\text{Ba}_2\text{Bi}_2\text{O}_5$	small, tenth's of mm	crystallized from melt of stoichiometric composition on hot wire loop	complete single crystal x-ray structure anal.	high-temp form, metastable at room temp.	A. Mignelli, A. Perloff A. Perloff	A. D. Mignelli, A. Perloff and G. Flock, "The Crystal Structure of the High Temperature Form of Barium Borate, $\text{BaO}\cdot\text{B}_2\text{O}_5$ ", Acta Cryst., <u>20</u> , 819 (1966).
$\text{Bi}_4\text{K}_2\text{O}_4 \cdot \text{Ba}_2\text{O}_3$	~ 0.1 mm range	crystallized from melt of stoichiometric composition	complete single crystal x-ray structure anal.	complete single crystal x-ray structure anal.	A. Perloff	A. Perloff, paper in prep. for submission to Acta Cryst.
$\text{PbO} \cdot 2\text{Ba}_2\text{O}_3$	~ 0.1 mm range	crystallized from melt of stoichiometric composition	unit cell and diffraction symbol from single crystal data	structure by analogy to isomorphous $\text{SrO} \cdot 2\text{Ba}_2\text{O}_3$	A. Perloff	A. Perloff and S. Block, "The Crystal Structure of the Strontium and Lead Tetraborates", $\text{SrO} \cdot 2\text{B}_2\text{O}_3$ and $\text{PbO} \cdot 2\text{B}_2\text{O}_3$ ", Acta Cryst., <u>20</u> , 274 (1966).
$\text{Ba}_4\text{O}_7\text{Pb}$	needles; ~ 0.1 mm length	crystallized from melt of stoichiometric composition	complete single crystal x-ray structure anal.	see $\text{Ba}_4\text{O}_7\text{Pb}$	A. Perloff	See ref. under $\text{Ba}_4\text{O}_7\text{Pb}$.
$\text{SrO} \cdot 2\text{Ba}_2\text{O}_3$	~ 0.1 mm, cross-section	~ .01 mm	melt solidification, required, 1 x 1 x 2 mm	spectrochemical anal.; optical, x-ray powder diffractometry (from ambient to 850°C); differential thermal anal.	C. R. Robbins E. M. Levin	C. R. Robbins and E. M. Levin, "Phase Transformation in Barium Tetraborate", J. Res. NBS, <u>73A</u> , 615 (1969).
$4\text{Ba}_2\text{O}_3 \cdot \text{BaO}$ $\text{BaB}_8\text{O}_{13}$	small (~ 0.1 mm range)	twinned crystal, 1 x 1 x 2 mm	seeding for crystallization	piezoelectric at ambient temp. (orthorhombic form); transforms reversibly to tetragonal form at 700°C	A. Perloff	high temp. form, metastable at room temp.
$4\text{Ba}_2\text{O}_3 \cdot \text{Na}_2\text{O}$ $\text{Na}_2\text{O} \cdot 4\text{Ba}_2\text{O}_3$		crystallized from melt of stoichiometric composition on hot wire loop	complete single crystal x-ray structure anal.		A. Hyman, A. Perloff, F. Mauer, and S. Block, "The Crystal Structure of Sodium Tetraborate", Acta Cryst., <u>22</u> , 815 (1967).	

$\text{Ba}_2\text{Ge}_4\text{O}_9$	1×1 $\times 3$ mm	melt solidification of stoichiometric composition	optical and x-ray diffractionometry (1), including single crystal structure detm. (2)	type structure for AB_2O_9 germanates, see also $\text{Ge}_4\text{O}_9\text{Pb}$ and $\text{Ge}_4\text{O}_9\text{Sr}$ in SECTION V	C. R. Robbins	(1) C. R. Robbins and E. M. Levin, "Tetragermanates of Strontium, Lead and Barium of Formula Type AB_2O_9 ", J. Res. NBS, <u>65A</u> , 127 (1961). (2) C. Robbins, A. Perloff, and S. Block, "Crystal Structure of $\text{Ba}_2\text{Ge}_4\text{O}_9$ and its Relation to Benitoite", J. Res. NBS, <u>70A</u> , 385 (1966).
$\text{Ba}_2\text{Ge}_2\text{O}_5\text{Ti}$ $\text{Ba}_2\text{TiGe}_2\text{O}_8$	1×1 3 mm	melt solidification and melt solidification using TiO_2 as flux	spectrochemical; optical and x-ray powder diffractometry; differential thermal anal.	crystals exhibit pronounced domain structure, show very strong piezoelectric response (1); phosphor (2)	C. R. Robbins	(1) Work in progress with particular interest in the crystal chemistry, domain structure and piezoelectric property of the compound. (C. R. Robbins, 1969). (2) G. Blasse, "Fluorescence of Compounds with Fresnoite ($\text{Ba}_2\text{Si}_2\text{O}_8$) Structure", J. Inorg. Nucl. Chem., <u>30</u> , 2283 (1968).
$\text{Ba}_2\text{O}_8\text{Si}_2\text{Ti}$ $\text{Ba}_2\text{TiSi}_2\text{O}_8$	1×1 5 mm	melt solidification using TiO_2 as flux	spectrochemical; optical, x-ray powder diffractometry (powder and single crystal), differential thermal anal.	piezoelectric (1); phosphor (2); type structure	C. R. Robbins	(1) C. R. Robbins, "Growth of Fresnoite ($\text{Ba}_2\text{Si}_2\text{O}_8$) From a TiO_2 Flux and Relation to the System $\text{BaTiO}_3 - \text{SiO}_2$ ", J. Res. NBS, <u>74A</u> , 229 (1970). (2) See ref. under $\text{Ba}_2\text{Ge}_2\text{O}_8\text{Ti}$.

$\text{Be}_2\text{Cu}\text{Li}_2\text{Si}_2\text{Y}$	light blue, 1 mm; from $\text{Na}_2\text{W}_2\text{O}_7$, also studied as poly-crystalline material	slow cooling from $\text{Na}_2\text{W}_2\text{O}_7$, or $\text{Na}_2\text{W}_2\text{O}_7$ flux	precession and x-ray powder diffraction anal.	see under $\text{Be}_2\text{LnMo}_1\text{Si}_2$ (M = Mg, Ni, Zn, Co, Cu, Fe, Mn, Cd), SECTION V	H. S. Peiser	J. Ito, "The Synthesis of Gado- linite", Proc. Jap. Acad., <u>41</u> , 404 (1965).
$\text{Cu}_2\text{Be}_2\text{Si}_2\text{O}_9$ copper- yttrium gadolinite	bright blue, 2 mm doped with V^{+4}	slow cooling from V_2O_5 or $\text{Ba}_3(\text{VO}_4)_2$ flux	precession and x-ray powder diffraction anal.	see also Bi_2GeO_4	C. Frondel and J. Ito, "Synthesis of the Scandium Ana- logues of Beryl", Amer. Mineral., <u>53</u> , 943 (1968).	C. Frondel and J. Ito, "Synthesis of the Scandium Ana- logues of Beryl", Amer. Mineral., <u>53</u> , 943 (1968).
$\text{Bi}_{12}\text{GeO}_{10}$ $\text{GeBi}_{12}\text{O}_{10}$		see $\text{Bi}_{12}\text{Mo}_{20}$	optical	see $\text{Bi}_{12}\text{Ga}_2\text{O}_{15}$ A. Feldman D. Horowitz	H. S. Peiser	E. L. Venturini, E. G. Spencer and A. A. Bailman, "Elasto- optic Properties of $\text{Bi}_{12}\text{Ge}_{20}\text{O}_{10}$, $\text{Bi}_{12}\text{Si}_{20}\text{O}_{10}$, $\text{Sr}_{1-x}\text{Nb}_{2-x}\text{O}_6$, J. Appl. Phys. , <u>40</u> , 1622 (1969).
$\text{Bi}_{12}\text{Mo}_{20}$ $6\text{Bi}_2\text{O}_3 \cdot \text{Mo}_2$	M = Ge, Si, Ti; 0.5 x 1 cm	melt grown	Faraday rotation; piezoelectric electro-optic	see also Bi_2GeO_4	W. S. Brower	E. L. Venturini, E. G. Spencer and A. A. Bailman, "Elasto- optic Properties of $\text{Bi}_{12}\text{Ge}_{20}\text{O}_{10}$, $\text{Bi}_{12}\text{Si}_{20}\text{O}_{10}$, $\text{Sr}_{1-x}\text{Nb}_{2-x}\text{O}_6$, J. Appl. Phys. , <u>40</u> , 1622 (1969).
$7\text{Bi}_2\text{O}_3 \cdot \text{ZnO}$ $17\text{Bi}_2\text{O}_3 \cdot \text{Ga}_2\text{O}_3$	0.5 x 1 cm 0.5 x 1 cm	melt grown melt grown	optical	pulled from melt, prep. by W. S. Brower	W. S. Brower	A. Feldman D. Horowitz optically active, high Faraday ro- tation; see also $\text{Bi}_{12}\text{Ge}_{12}\text{O}_{40}$

Br_2	bromine	from liquid at 20°C, 5Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	C. E. Weir, G. J. Piermarini, and S. Block, "Crystallography of Some High Pressure Forms of $\text{C}_6\text{H}_5\text{CS}_2$, Br_2 , CCl_4 , and KNO_3 ", <i>J. Chem. Phys.</i> , <u>50</u> , 2089 (1969)
CCl_4	I structure	from liquid at 20°C, 1Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	See ref. under Br_2 .
	II structure	from CCl_4 -I at 80°C, 8Kb				
	III structure	from CCl_4 -I at 120°C, 10Kb				
CS_2		frozen from liquid at 20°C, 12Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	See ref. under Br_2 .
$\text{C}_5\text{FeN}_6\text{Na}_2\text{O} \cdot 2\text{H}_2\text{O}$		1 x 1 x 3 in.	controlled growth from aq. soln.	Mossbauer spectroscopy	A. T. Horton Standard Reference Material #725; Mossbauer spectroscopy	J. J. Spijkerman, D. K. Snediker, F. M. Ruegg, and J. R. Devee, "Mossbauer Spectroscopy Standard for the Chemical Shift of Iron Compounds", <i>NBS Misc. Publ.</i> , 260-13 (1967).
$\text{Na}_2\text{Fe}(\text{CN})_5\text{NO} \cdot 2\text{H}_2\text{O}$						"Standard Reference Material for Mossbauer Spectroscopy", <i>NBS Tech. News Bull.</i> , <u>50</u> , 73 (May 1966).
C_6H_6	I structure, see also SECTION II	frozen from liquid at 20°C, 0.6 Kb	x-ray diffractometry	unit cell; space group; phase equilibrium	S. Block G. J. Piermarini C. E. Weir*	See ref. under Br_2 .

$C_{10}H_{10}Cl_2CuN_2O_2$	$1 \times 1 \times 1$ to $5 \times 5 \times 5$ mm	grown from methanol soin.	optical and EPR spectrometry; chemical anal.	metal-metal interaction study	G. F. Kokoszka R. W. Duerst and G. F. Kokoszka, "Hyperfine Fields in Dimeric Coordination Complexes," J. Chem. Phys., 51, 1673 (1969).
$(C_5H_5NO)_2Cl_2$ with the following dopants: Ba, Cd, Ni, In (0.1-%), Pb, and Zn bis(pyridine-N-oxide copper (II) chloride	1 x 4 in $(C_5H_5)_2Fe$ ferrocene	melt solidification	infrared and Raman spectrophotometry	infrared and Raman spectra vibrational transitions	A. T. Horton A. D. Mighell, C. W. Reimann, and F. A. Mauer, "The Crystal and Molecular Structure of Di aquo- bis(2,2'-Bimidazole) Nickel (II) Dinitrate, $Ni(C_6H_6N_4)_2(H_2O)^2$ (NO ₃) ₂ , Acta Cryst., B25, 60 (1969).
$C_{12}H_{12}N_{10}NiO_6 \cdot 2H_2O$ $Ni(C_6H_6N_4)_2$ $(H_2O)_2(NO_3)_2$		$C_6H_6N_4$ (2,2'-bi-imidazole) + $Ni(NO_3)_2$ + H_2O	single crystal x-ray anal.	study of hydrogen bonding be- tween co- ordinated water and bimidazole molecules with nitrate groups	C. W. Reimann A. D. Mighell, C. W. Reimann, and F. A. Mauer, "The Crystal and Molecular Structure of Di aquo- bis(2,2'-Bimidazole) Nickel (II) Dinitrate, $Ni(C_6H_6N_4)_2(H_2O)^2$ (NO ₃) ₂ , Acta Cryst., B25, 60 (1969).
$C_{12}H_{16}Br_2N_8Ni$		$C_3H_4N_2$ (pyrazole) + $NiBr_2$ + H_2O	x-ray anal. single crystal structure detm.: optical spectrophotometry	role of internal hydrogen bond in detm. elec- tronic energy levels of Ni ²⁺	C. W. Reimann A. D. Mighell, C. W. Reimann, and F. A. Santoro, "The Crystal and Molecular Structure of Di- pyrazole-nickel Chloride, $Ni(C_3H_4N_2)_4Cl_2$ ", Acta Cryst., 23, 135 (1967).
$(C_3H_4N_2)_4NiCl_2$ tetraakis (pyrazolo)- nickel (II) bromide		$(C_3H_4N_2)_4NiCl_2$ + H_2O			

$C_{12}H_{18}$	$1''$ dia. \times $3''$	melt solidification $C_6(CH_3)_6$ hexamethylbenzene	x-ray, neutron diffractionometry; differential thermal anal.	crystal structure; phase transitions; lattice dynamics	A. T. Horton
$C_{12}H_{18}N_4Ni_3O_6 \cdot H_2O$ $[(H_2O)^3(C_2H_5N_2)_2 \cdot Ni]^2 \cdot Ni(NO_3)_6(H_2O)_2$		$C_3H_4N_3(1,2,4\text{-triazole})_2 \cdot Ni(NO_3)_2 \cdot H_2O$	single crystal x-ray anal.	optical and magnetic properties of polynuclear species	C. W. Reimann
$C_{12}H_{18}N_4Ni_4O_6$ $(C_2H_4N_2)_6Ni(NO_3)_2$		$C_3H_4N_2(\text{pyrazole})_2 \cdot Ni(NO_3)_2 \cdot H_2O$	single crystal x-ray anal.	study of competition between hydrogen bonding and coordinate bonding in cationic packing	C. W. Reimann
$C_{18}H_{24}MN_4O_6$		$M = Cd, Co,$ $(C_3H_4N_2)_6Cd(NO_3)_2$ $(C_3H_4N_2)_6Co(NO_3)_2$ $(C_3H_4N_2)_6Ni(NO_3)_2$	$C_3H_4N_2(\text{imidazole})_2 \cdot M(NO_3)_2 \cdot H_2O$	single crystal x-ray anal.	C. W. Reimann

C_2H_4	2" dia. x 4"	melt solidification n-C ₂ H ₄ n-eicosane	electrical, optical ab- sorption measurements	detr. free carrier mobility and G values; radiolysis measurement by use of optical ab- sorption techniques	A. T. Horton
CaF_2	1 x 3 x 5 mm	commercially obtained; cry- stals annealed in He + HF at 500-1200°C, rapidly quenched	absorption (200 to 2500 nm), EPR spectrometry; chemical anal. for Gd and accidental impurities	controlled point-defect equilibrium studies; oxygen free	A. Franklin
also Gd-doped (0.1 to 1.0%)					A. Franklin and S. Marullo, "Orientation Kinetics of Gd ³⁺ F- Interstitial Gains in CaF ₂ ", J. Physics C, British Ceramic Soc., London, Dec. 1969, in press.
$CaGeO_5$	1 x 1 x 2 mm	melt solidi- fication from stoichiometric composition	spectrochemical anal.; optical, x-ray diffracto- metry (powder and single crystal)	isostructural with synthetic CaO_3SiTi	C. R. Robbins
$CaMoO_4$		see under MoO_4			C. R. Robbins, "Synthetic $CaTiSiO_5$ and its Germanium Analogue ($CaTiGeO_5$)", Mat. Res. Bull., 3, 693 (1968).

$\text{Ca}_0.5\text{Si}_1$	1×2.5 $\text{cm} \times (1)$	melt solidification (2), and Czochralski growth (1)	spectrochemical anal.; optical, x-ray diffracto- metry [powder and single crystal (2)]	W. S. Brower and C. R. Robbins, "Growth of CaTiSiO_5 by the Czochralski Method", <i>J. Cryst. Growth</i> , <u>3</u> , (1969).
CaTiSiO_5	0.5×0.5 $\times 1.5 \text{ cm}$ (2)	see under CaW_4W		(2) See ref. under CaGeO_3Ti

CaO_4W		see under Mo_4W		C. R. Robbins type structure W. S. Brower for pure CaTiSiO_5 (space group $P\bar{2},n$); mineral with imparities, $\text{CaTiSiO}_4(\text{OH},$ $\text{F})$, has symmetry C_2/c ; see also CaGeO_3Ti
$\text{Ca}_3\text{Fe}_2\text{Mo}_{12}\text{Si}_2$		M = Ti,Zr	slow cooling of Li_2MoO_4 flux	W. S. Peiser crystallographic sites detn. for both Ti and Zr in garnet structure
$\text{Ca}_3\text{TiFe}_2\text{Si}_2\text{O}_1$			titanium garnet, dark brown, 2 mm also poly- crystalline	J. Ito and C. Frondel, "Synthetic Zirconium and Titan- ium Garnets", Amer. Mineral., <u>52</u> , 773 (1967).
$\text{Ca}_3\text{ZrFe}_2\text{Si}_2\text{O}_1$			zirconium garnet, chestnut brown, 0.55 mm, also polycrystal- line	

CdF_2	1 cm	Briagman growth	electrical	W. R. Hosler W. S. Brower
also with dopings				p. Eisenberger and P. S. Pershan, "Electron Spin-Resonance and Infrared Studies of Semiconducting, Rare-Earth-Doped CdF_2 ", <i>Phys. Rev.</i> , <u>167</u> , 292 (1968).

(1) Cu
CuCl

Vapor and
Bridgeman
growth; prep'd.
by D. E.
Roberts, also
supplied by
A. F. Armington
AFCRL, Bedford,
Mass., and by
A. Linz, Mass.
Inst. Tech.,
Cambridge, Mass.

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NBS Tech. Rpt. 10013.

optical, dielectric
modulator

A. Feldman
D. Horowitz

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Horowitz, "Refractive
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Opt. Soc. Am.* 59,
1406 (1969).

ClO_3Na
 NaClO_3

optical, dielectric
modulator

A. Feldman
D. Horowitz

R. A. Forman
measurement
of quadrupole
coupling const.

M. Linzer
for D on ND_4^+
configuration;
torsional motion
of ND_4^+ ; crystal
structure.

M. Linzer and R.
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4690 (1967).

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R. Brooks, A. T.
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Torgesen, "Occlusion
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Solution-Grown
Crystals", *J. Cryst.
Growth*, 2, 279
(1968).

TlCl	~ 2 mm, very soft	Bridgeman growth; prep'd. by D. E. Roberts	optical luminescent properties	A. Feldman L. H. Grabner	R. Z. Bachrach and F. C. Brown.
also with dopants Cd, Pb, Nd, T					"Exciton Structure of TlCl and TlBr", Phys. Rev. Letters, 21, 685 (1968).
C ₁ Tl					A. Feldman, "Anoma- lous Splitting of the Exciton Peak in the Reflectivity of TlCl and TlBr", Bull. Am. Phys. Soc., 14, 428 (1969).
					L. H. Grabner, "Edge Fluorescence in TlBr", Bull. Am. Phys. Soc., 14, 131 (1969).
C ₁ Cl ₃ CsPb CsPbCl ₃	yellow, 1 cm. dia. x 2 cm.	grown by W. S. Brower, Bridgeman method	dielectric constant	K. F. Young	C. K. Noller, "Structure of Perovskite-Like Ce Pb Trihalides", Kgel. Danske Videnskab. Selskab, Mat. Fys. Medd., 32, 1 (1959); also see Nature, 182, 1436 (1958).
C ₁ Li ₆ K ₂ Re K ₂ ReCl ₆	\sim mm size	controlled evaporation, from soln.	ultra-sonic attenuation	A. T. Horton	H. S. Peiser
CoM ₂ /Si ₂ Ca ₂ CoSi ₂ O ₇ Sr ₂ CoSi ₂ O ₇	M = Ca, Sr; dark blue; 2 mm. Ca- and Sr cobalt akermanite; also polycrystalline analogues with M = Cd, Cu, Fe, Mg, and Mn	slow cooling, flux Na ₂ WO ₄	x-ray powder diffractometry	J. Ito and H. S. Peiser, "Distorted Tetrahedra in Strontium Copper Akermanite", J. Res. NBS, 73A, 69 (1969).	

$\text{CrH}_6\text{Mo}_6\text{Na}_4^{0.24}\cdot\text{H}_2\text{O}$	n = 8, aq. soln. of Na ₂ Mo ₆ ⁴⁺ ·2H ₂ O + Cr(NO ₃) ₃ ·H ₂ O at pH = 4.5 prepn. leads to mm size crystals, no comm- ercial supply available	complete single crystal x-ray structure anal.	A. Perloff readily dehydrates to a con- position approximating (Na ₃ CrMo ₆ O ₂₄ H ₆) · 2H ₂ O
$\text{Na}(\text{CrMo}_6\text{O}_{24}\text{H}_6)\cdot\text{H}_2\text{O}$	n = 13, aq. soln. of Na ₃ CrMo ₆ O ₂₄ H ₆ at pH = 4.5 prepn. leads to mm size crystals, no commercial supply available	crystallizes on evaporation of aq. soln. of octahydrate at room temp.	readily dehydrates to a lower hydrate
$\text{CrH}_4\text{NO}_8\text{S}_2 \cdot 12\text{H}_2\text{O}$	from aq. soln. $\text{NH}_4\text{Cr}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	optical, magnetic	G. A. Candela S. E. Stokowski see also $\text{Al}_{n-1}\text{Cr}_n\text{O}_8\text{S}_2 \cdot 12\text{H}_2\text{O}$
$\text{CuO}_4\text{S} \cdot 5\text{H}_2\text{O}$	1 cm ²	controlled evaporation from soln.	NMR spectrometry A. T. Horton
$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$			NMR spectro- scopy, wave guide experiments

Cu_2O 1.5 x
 6 cm
grown from melt

W. S. Parker optical and electrical measurements;
 H. S. Parker air sensitive

H. S. Parker , "Melt Cuprous Oxide Single Crystals", Abstracts, ACCG-NBS Conf. on Crystal Growth, August 11-13, 1969, Washington, D. C., p. 115.

prep'd. by
 W. S.
Brower

electrical

R. A. Forman
 W. R. Hosler
 K. F. Young



M = Gd
La
Nd
Pr
Sm

largest crystal
2 x 2 x
6 cm
colorless
purple
green
yellow

x-ray powder diffraction anal.; visible emission spectrometry

W. S. Brower , "Silicate Apatites and Oxygenapatites", Amer. Mineral., 53, 890 (1968).

33

F_2Mg $\sim 1 \text{ cm}^3$
 MgF_2

pulled from melt, D. E. Roberts

optical; EPR spectrometry

H. S. Peiser
 W. R. Hosler
 K. F. Young

oxide-semiconductor dielectric constant

R. A. Forman
 W. R. Hosler
 K. F. Young

W. S. Brower , "Growth of Lead Fluoride Crystals from the Melt", Proc. Phys. Soc., 68B, 165 (1955).

F_2PbPbF_2
Bridgeman growth
also Y, rare-earth dopings

W. R. Hosler
 W. S. Brower

Bridgeman growth

D. A. Jones , "Growth of Lead Fluoride Crystals from the Melt", Proc. Phys. Soc., 68B, 165 (1955).

H_2O - VII ice VII	structure VII X = Br, Cl, I also with Tl doping	from liquid at $100^\circ C$ and 30 Kb	x-ray diffracto- metry	unit cell, space group, phase equi- librium	C. E. Weir, S. Block, and G. Piermarini "Single Crystal X-ray Diffraction at High Pressures", J. Res. NBS, <u>69C</u> , 275 (1965).
H_4NX	$\sim 1\text{ cm}^3$	from aq. soln. by evaporation	optical	relation to R. A. Forman alkali halides	R. D. Deslattes, J. L. Torgesen, B. Paretskin, and A. T. Horton, "Preliminary Studies on the Characterization of Solution-Grown ADP Crystals", Adv. in X-ray Anal., <u>8</u> , p. 315 (1965).
H_6NO_4P	$3/4 \times 3 \text{ in}$	controlled growth from soln.	optical microscopy (etch pits); x-ray topography	monochro- meters, electro- optics, x-ray reference crystals	R. D. Deslattes, J. L. Torgesen, B. Paretskin, and A. T. Horton, "Observation of Dislocations in ADP: Production of Dislocation-Free Crystals", J. APPL. Phys., <u>37</u> , 541 (1966).
$InNa_6Si_2$ $NaInSi_2O_6$	colorless indium aspirine, 1 cm; also obtained as polycrystal- line	slow cooling from Na_2WO_4 or Na_2MoO_4 flux	powder and precession x-ray diffraction anal.	see also $Na_0.6ScSi_2$	J. Ito, "Synthetic Indium Silicates and Hydrogarnet", Am. Mineral., <u>53</u> , 1663 (1968).

KNO ₃	III	structure III	obtained from KNO ₃ II at elevated temp. and pressure.	x-ray diffracto- metry	detm. unit cell, space group, phase equilibrium	S. Block G. Piermarini C. E. Weiss	See ref. under Br ₂ .
KNO ₃	IV	structure IV	from satu- rated aq. sln. KNO ₃ at 100°C, 20 Kb				
KO-Ta			prepd. by W. S. Brower; also obtained with doping from A. Linz, Mass. Inst. Tech., Cambridge, Mass.; flux growth	electrical, optical oxide semiconductor	M. I. Cohen W. R. Hosler	M. I. Cohen, et al., "Lattice Absorp- tion in Strontium Titata- nate", Bull. Am. Phys. Soc., <u>14</u> , 498 (1969).	W. R. Hosler and H. P. R. Frederiks, "Magnetoresistive Effects in KtaO ₃ ", Sol. State Comm., <u>7</u> , 1443 (1969).

$M = Ca_{1-x}Fe_x$	see also Mn_xCo_{1-x}			
$CaMoO_4$	x-ray topography; dielectric constant; etch pits	acousto-optic devices	W. S. Brower	W. S. Brower and P. H. Fang, "Dielectric Constants of $PbMoO_4$ and $CaMoO_4$ ", Phys. Rev., <u>119</u> , 646 (1967).
$1 \times 3 \text{ cm}$	melt grown		J. B. Wachtman, Jr., W. S. Brower, E. M. Farabaugh, "Elastic Constants of Single Crystal $CaMoO_4$ ", J. Am. Ceram. Soc., <u>51</u> , 341 (1968).	W. S. Brower and P. H. Fang, "Dielectric Constants of $PbMoO_4$ and $CaMoO_4$ ", J. Appl. Phys., <u>38</u> , 2391 (1967).
$SrMoO_4$		elastic constants of $CaMoO_4$		
	mechanical			
MO_4W	$M = Ca, Pb,$ $Sr, Zn;$ $1 \times 3 \text{ cm}$	melt grown	x-ray topography; dielectric constant; etch pits	W. S. Brower See refs. under $MMoO_4$.
$CaWO_4$				
$PbWO_4$				
$SrWO_4$				
$ZnWO_4$				
$ZnWO_4$	1 cm	optical	magneto-optical properties	R. F. Blunt

MnMoO ₄	1 cm	pulled from melt; prepd. by D. E. Roberts	optical	magneto-optical properties	R. F. Blunt	See also under MnWO ₄ and Mo ₄ W for additional refs. to Scheelite crystals.
MnO ₄ W	1 cm	pulled from melt; prepd. by D. E. Roberts.	optical	magneto-optical properties	R. F. Blunt	See also Mo ₄ W.
MoO ₄ Pb MoO ₄ Si ₂	see MMoO ₄					
Na _{0.6} ScSi ₂ NaScSi ₂ O ₆	colorless, scandium aegirine, 0.5 cm; also as poly- crystalline	slow cooling from Na ₂ WO ₄ or Na ₂ MoO ₄ flux	powder and pre- cession x-ray diffraction anal.	see also InNaO ₆ Si ₂	H. S. Peiser	J. Ito and C. Frondel, "Synthesis of Scandium Analogues of Aegirine, Andradite and Melano-tekite", <i>Am. Mineral.</i> , <u>53</u> , 1276 (1968).
NiTi	wire	obtained from NOL, White Oak, Md.	electrical and magnetic	exhibits martinitic transition; i.e., shows mechanical memory through transition	W. R. Hosler	F. E. Wang, B. F. DeSavage, W. J. Buehler, and W. R. Hosler, "The Irreversible Critical Range in the TiNi Transition", <i>J. Appl. Phys.</i> , <u>39</u> , 2166 (1968).
TiNi	2 × 1 × 1 cm	selected from arc melt by G. R. Findlay, Norton Co.	Laue diffraction	elastic constants	J. Wachtman, Jr. P. B. Maceado, W. Capps, and J. B. Wachtman, Jr., "Elastic Constants of Single Crystal ThO ₂ at 25°C", <i>J. Am. Ceram. Soc.</i> , <u>47</u> , 651 (1964).	
O ₂ Th ThO ₂						

O_2U	$3 \times 1 \times 1$ cm	selected from arc melt by H. J. Anderson of Hanford Atomic Products Operation	density, coulometric titration, spec. analysis, Laue diffraction	elastic constants	J. Wachtman, Jr. M. L. Wheat, H. J. Anderson and J. L. Bates, "Elastic Constants of Single Crystal UO_2 at $25^\circ\text{C}"$, J. Nuclear Materials, <u>16</u> , 39 (1965).
PbSiO_3	O_3PbSi	alamosite, colorless, 2 mm; also prep. as polycrystal- line	hydrothermal, spontaneous nucleation isothermal	x-ray structural anal. (M. Mydlar, Ceramic Engr. Dept., Univ. of Mo.)	H. S. Peiser J. Ito, "Synthesis of Some Lead Calcium Zinc Silicates", Amer. Mineral., <u>53</u> , 231 (1968).

SrTiO_3
Sr TiO_3 , pure,
also with
dopants:
 $\text{Al}, \text{Ga}, \text{Nb}$

flame fusion;
also supplied
by D. Beals,
National Lead
Co., South
Amboy, N. J.

optical, electrical;
EPR spectrometry
by D. Beals,
National Lead
Co., South
Amboy, N. J.

insulator;
semiconductor, M. I. Cohen
superconductor; A. Feldman
pseudo-ferroelectric; A. H. Kahn
photochromic

A. H. Kahn and A.
J. Leyendecker,
"Electronic Energy
Bands in Strontium
Titanate", Phys.
Rev., 135, A1321
(1964).

H. Chang
A. Feldman
L. H. Grabner
W. R. Hosler
A. H. Kahn

T. Chang
M. I. Cohen
A. Feldman
L. H. Grabner
W. R. Hosler
A. H. Kahn

H. P. R. Frederikse
W. R. Hosler, and W.
R. Thurber, "Experi-
mental Evidence
Concerning the
Conduction and
Band of SrTiO_3 ",
J. Phys. Soc. Japan,
21, Suppl., 32
(1966).

J. F. Schooley and W.
R. Thurber, "Super-
conductivity in
Semiconducting
 SrTiO_3 ", J. Phys.
Soc. Japan, 21,
Suppl., 639 (1966).

A. Feldman and D.
Horowitz, "Stress-
Induced Dichroism
at the Absorption
Edge of Strontium
Titanate", Sol.
State Comm., 5, 607
(1968).

C. R. Robbins
"Growth
of Strontium Titanate
from a Silica Flux",
J. Cryst. Growth, 2,
402 (1968).

C. R. Robbins
"Absence of
fluorides,
etc."

prep. in
spectrochemical,
optical and x-ray
powder diffracto-
metry

SrTiO_3 1 - 2.5 mm

melt solidi-
fication using
 SiO_2 as flux

V. POLYCRYSTALS

NBS STAFF MEMBERS
TO BE CONTACTED
FOR ADDITIONAL
INFORMATION

REFERENCES (Author,
Title, Journal)

COMPOSITION	NATURE OF MATERIAL	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	NBS STAFF MEMBERS TO BE CONTACTED FOR ADDITIONAL INFORMATION	REFERENCES (Author, Title, Journal)
Ag	high-purity rod, 1/4 in. dia. x 2-1/2 in.	special prepn. for Office of Standard Reference Materials, SRM # 748	Knudsen, torque Knudsen, and mass spectrometric detm. of vapor pressure as function of temperature; detm. heat of sublimation	calibrn. of vapor pressure apparatus and techniques (10 ⁻⁸ to 10 ⁻³ atm., 1000-1600°K); this and other elements (see Al, Cd, Pt, and W) will each cover a different temperature range; these five materials cover 400-3000°C	R. C. Paule	R. C. Paule and J. Mandel, NBS Special Publication # 260.
41	"high-AgNO ₃ "	polycrystalline surface, $\alpha \sim 50\text{ }\mu$	high temp-erature x-ray diffractometer furnace	x-ray powder diffractometry	E. R. Planté	See ref. above.
AgNO ₃	100-300 mesh aluminum nitride	four commer-cial sources	x-ray diffractometry	unstable at room temp.	E. M. Levin	E. M. Levin, "X-Ray Determination of the Thermal Expansion of Silver Nitrate", J. Am. Ceram. Soc., 52, 53 (1969).
AlN	Y-Al ₂ O ₃ + BaCO ₃ at 1200°C for 1.5 hr.				F. E. Brinckman	
Al ₂ BaO ₄			x-ray diffractometry; petrography microscopy		H. Swanson J. deGroot H. McMurdie	H. Swanson et al., "Standard X-Ray Diffraction Patterns", NBS Monograph No. 25, Sect. 5, p. 11 (1967).

BH ₄ M KBH ₄ LiBH ₄ NaBH ₄	M = Li, K, Na; obesd. also in aq. soln.	commercial	NMR (¹ H, ¹¹ B) spectrometry	model com- pounds, destr. phase trans- itions and motions in solids	T. C. Farrar	T. C. Farrar and T. Tsang, "A Nuclear Magnetic Resonance and Relaxation Study of Dimethoxyborane", <i>J. Res. NBS</i> , <u>73A</u> , 195 (1965).
BO ₃ Sc ScBO ₃	~50μ part- icles, calcite type	solid state rxn. of Sc ₂ O ₃ + B ₂ O ₃ in sealed tube	x-ray powder diffractometry, polarizing microscopy	phase diagram	E. M. Levin	E. M. Levin, "System Sc ₂ O ₃ -B ₂ O ₃ ", <i>J. Am. Ceram. Soc.</i> , <u>50</u> , 53 (1967).
B ₂ O ₃		controlled dehydration of H ₃ BO ₃	compressibility to 10 Kb	See ref. under BH ₃ O [*] .	C. E. Weir*	See ref. under BH ₃ O [*] .
B ₂ O ₃ •3Nb ₂ O ₅ "Nb ₂ O ₅ •B ₂ O ₃ "	~50μ	solid state rxn. of B ₂ O ₃ + Nb ₂ O ₅ in sealed Pt tube	x-ray powder diffractometry	phase diagram	E. M. Levin	E. M. Levin, "Phase Equilibrium on the System Niobium Pentoxide-Boric Oxide", <i>J. Res. NBS</i> , <u>70A</u> , 11 (1966).
Borate minerals, anhydrous	~80 in number, fine powders	metal oxide + H ₃ BO ₃ at elevated temp.	infrared (2000- 300 cm ⁻¹) spectro- metry	correlation of anion and spectra	C. E. Weir*	C. E. Weir and R. A. Schroeder, "Infrared Spectra of the Hydrated Crystalline Inorganic Borates", <i>J. Res. NBS</i> , <u>68A</u> , 465 (1965).
Borate minerals, hydrated	~42 natural and synthetic	obtained from the Smithsonian Inst. U. S. Geological Survey, Wash- ington, D. C. and Menlo Park, Calif.	infrared spectro- metry, principally in 2000-300 cm ⁻¹ region	evaluation of limita- tions of spectra in identification, effects of treatment on spectra	C. E. Weir*	C. E. Weir, "Infrared Spectra of the Hydrated Borates", <i>J. Res. NBS</i> , <u>70A</u> , 153 (1966).
BaF ₃ Li LiBaF ₃		fuse stoichio- metric amt. LiF + BaF ₂ at 800°, annealed at 500° to re- move BaF ₂ ppt.	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 35.	

$\text{BaGe}_3\text{O}_9\text{Ti}$	20-40 u	solid state rxn. or $\text{BaTiO}_3 + \text{GeO}_2$ in sealed Pt tube at 1160°C	stable from 1132° to 1235°C but readily obtainable by quenching to ambient temp.; serves as an intermediate between BaGe_3O_9 and $\text{BaTiSi}_3\text{O}_9$	C. R. Robbins	C. R. Robbins, "The Compound $\text{BaTiSi}_3\text{O}_9$, J. Am. Ceram. Soc., 43, 610 (1960).
$\text{BaTiGe}_3\text{O}_9$				H. S. Peiser	J. Ito, "The Synthesis of Gadoilinite", Proc. Jap. Acad., 41, 404 (1965).
$\text{Be}_2\text{Cu}_0\text{Si}_2\text{Y}_2$		slow cooling of solute in Na ₂ WO ₄ or Na ₂ W ₂ O ₇ flux with Mn ₂ Be ₂ -S ₂ O ₁₀ , where M=Mg, Ni, Zn, Co, Cu, Fe, Mn, and Cr; single crystal (Cmma) also studied	precession and powder x-ray diffractometry	H. S. Peiser	J. Ito, "A Note on the Gadoilinite Synthesis", Proc. J. Acad., 42, 634 (1966).
$\text{Cu}_2\text{Be}_2\text{Si}_2\text{O}_1\text{O}$				J. Ito	J. Ito, "Synthesis of Calciogadolinite", Amer. Mineral., 52, 1523 (1967).
BiNbO_4		see also: Bi_2Ta $\text{Bi}_{10}\text{Ta}_4\text{V}$ $\text{Nb}_{10}\text{Ta}_4\text{Sb}_3$ $\text{O}_{14}\text{Sb}_4\text{V}$ $\text{O}_{14}\text{Sb}_4\text{V}$	solid state rxn. of $\text{Bi}_2\text{O}_3 + \text{Nb}_2\text{O}_5$ in sealed Pt tubes at $950 - 1250^\circ\text{C}$	R. S. Roth J. L. Waring	R. S. Roth and J. L. Waring, "Synthesis and Stability of Stibbotantalates, Chemically Similar ABO_6 Compounds", Amer. Mineral., 48, 1348 (1963).
BiTaO_4			solid state rxn. of $\text{Bi}_2\text{O}_3 + \text{Ta}_2\text{O}_5$ in sealed Pt tubes at $845 - 1150^\circ\text{C}$	R. S. Roth J. L. Waring	See ref. under BiNbO_4 .
BiVO_4			solid state rxn. of $\text{Bi}_2\text{O}_3 + \text{V}_2\text{O}_5$ in sealed Pt tubes at $500-1000^\circ\text{C}$	R. S. Roth J. L. Waring	See ref. under BiNbO_4 .

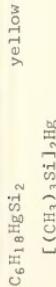
Bi_2O_3	cubic	high-temp. x-ray diffractometer furnaces; programmed heating and cooling	differential thermal anal.; x-ray diffracto- metry	obsd. at room temp. as metastable phase	E. M. Levin	E. M. Levin and R. S. Roth, "Polymorphism of Bismuth Ses- quioxide, I. Pure Bi_2O_3 ", J. Res. NBS, 68A, 189 (1944); <i>ibid.</i> , II, 197-206.
$\text{Br}_2\text{O}_2\text{Pb}_3$ $\text{Pb}_3\text{O}_2\text{Br}_2$		heat stoichio- metric PbO (red) + PbBr_2 in Au boat, in vacuo, at 350°C for 45 min.	x-ray diffracto- metry, petrographic microscopy	H. Swanson deGroot H. McMurdie	See ref. under Al_2BaO_4 , p. 32.	J. M. Sherfey, "Titanium-(III) Chloride and Titanium (III) Bromide", Inorg. Synth., 6, 57 (1960).
Br_3Ti TiBr_3		dark purple, fine powder, or small crystals; see also Cl_3Ti	elemental anal. for Ti, Br	sublimes at ~ 500°C with dis- proportionation, strong re- ducing agent; catalyst for olefin Poly- merization; air/ moisture sensitive	R. B. Johannessen J. M. Sherfey, "Titanium-(IV) Bromide", Inorg. Synth., 9, 46 (1967).	R. B. Johannessen G. L. Gordon, "Titanium (IV) Bromide", Inorg. Synth., 9, 46 (1967).
Br_4Ti		yellow, m.p. 38°C	$\text{TiCl}_4 + \text{HBr} \rightarrow$ at b.p. of $\text{TiCl}_{4-n}\text{Br}_n$	elemental anal., m.p., b.p.	strong Lewis Acid properties, air/moisture sensitive	R. B. Johannessen R. B. Johannessen and G. A. Canfield, "Magnetic Susceptibility and Dilution Effects in Low-Spin d^4 Complexes: Osmium (IV)", Inorg. Chem., 2, 67 (1963).
$\text{Br}_6\text{H}_8\text{N}_2\text{O}_8$ $(\text{NH}_4)_2\text{OsBr}_6$		mixed crystals (solid soln.) of $(\text{NH}_4)_2$ $(\text{Os-Pt})_6$,	(a) $\text{OSO}_4 +$ HBr(aq) (b) (a) + NH_4Br	elemental anal., x-ray diffracto- metry	parent com- pounds for magnetic susceptibility studies	R. B. Johannessen R. B. Johannessen and G. A. Canfield, "Magnetic Susceptibility and Dilution Effects in Low-Spin d^4 Complexes: Osmium (IV)", Inorg. Chem., 2, 67 (1963).

C_2Si	cubic, ~200 mesh	Si + C above m.p. of Si	x-ray diffracto- metry, vapor pressure	other poly- morphs pre- sent, C- contaminated	E. R. Plante
$\text{C}_2\text{Na}_2\text{O}_4$	NBS stand- ard sample	NBS stand- ard sample No. 10d	assay 99.9% $\text{Na}_2\text{C}_2\text{O}_4$; x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	H. Swanson, et al., "Standard X-Ray Diffraction Powder Patterns", NBS Monograph No. 25, Sect. 6, p. 70 (1968).
C_3Al_4	large cry- stallites embedded in polycrystal- line matrix	Al(1) + C at 1400°C	x-ray diffracto- metry, vapor pressure	usually contains excess C; moisture sensitive	E. R. Plante and C. H. Schreyer, "Dissoci- ation Pressure of Aluminum Carbide Using a Rotating Knudsen Cell", J. Res. NBS, 70A, 253 (1966).
C_4HCOO_4 $\text{HCo}(\text{CO})_4$	unstable liquid, studied as solid	see SECTION II	see SECTION II	see SECTION II	T. C. Farrar See SECTION II.
$\text{C}_4\text{H}_12\text{F}_2\text{O}_4\text{W}$ $(\text{CH}_3\text{O})_4\text{WF}_2$	colorless crystals m.p. 81-83°C	$\text{WF}_6 +$ $4\text{CH}_3\text{Si}(\text{CH}_3)_3$ in C_6F_6	NMR ($^1\text{H}, ^1\text{F}$) mol. wt. elemental analysis	one of a series of stable tungsten (VI) compounds; easily puri- fied by subli- mation; cis- difluoro- configuration; provides basis for W(VI) stereo- chemistry; air/ moisture sensitive.	F. E. Brinckman L. B. Handy, F. E. Brinckman, L. B. Handy*, "Chemistry of the Methoxyfluoro- tungsten (VI) Series", Chem. Comm., 214 (1970).

$C_6H_{10}CuO_4 \cdot H_2O$
Copper (II)
monohydrate

also Zn-doped
propionate monohydrate

heat $Cu(Zn)CO_3$
+ $[CH_3CH_2CO]_{20}$
+ H_2O
alkanoates from
EPR studies of
polycrystalline
samples



yellow
 $(CH_3)_3SiX +$
Na/Hg at 55°C
($X=Cl, Br$) (1)

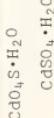
EPR spectrometry
model compounds for
detr. magnetic
parameters in
dimeric Cu(II)
alkanoates from
EPR studies of
polycrystalline
samples

$\text{CaCl}_{1.5}\text{S}$	$\text{CsCl} + \text{CaCl}_2$, at $\sim 900^\circ\text{C}$.	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4 , p. 21.
CsCdCl_3				
$\text{CaNa}_2\text{O}_8\text{S}_2$ $\text{Na}_2\text{Ca}(\text{SO}_4)_2$	glauberite $\text{CaCl}_2 + \text{Na}_2\text{SO}_4$ in aq. soln.; intermediate CaSO_4 forms, desired product after several hr., washed with alcohol	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $\text{C}_2\text{Na}_2\text{O}_4$, p. 59.
Cd	high purity rod, 1/4 in. dia. $\times 2\text{-}1/2$ in. Materials; SRM # 746	special prepn. for Office of Standard Reference Materials; SRM # 746	see Ag temp. range 400-600°K, see also under Ag	R. C. Paule See ref. under Ag*
CdCl_3Cs CsCdCl_3		stoichiometric $\text{CsCl} + \text{CdCl}_3$ fusion	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie
$\text{CdCl}_3\text{H}_4\text{N}$ NH_4CdCl_3		stoichiometric $\text{NH}_4\text{Cl} + \text{CdCl}_3$ (aq. soln.) + ppt.	x-ray diffracto- metry, petrographic microscopy	See ref. under Al_2BaO_4 , p. 19.
CdCl_3K KCdCl_3	fuse $\text{KCl} +$ $\text{CdCl}_3 \cdot 2.5\text{H}_2\text{O}$ at 550°C	fuse $\text{KCl} +$ $\text{CdCl}_3 \cdot 2.5\text{H}_2\text{O}$ at 550°C	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie
CdCl_3Rb	orthorhombic tetragonal	ppt. from aq. soln. $\text{RbCl} +$ CdCl_3 fuse $\text{RbCl} +$ CdCl_3 (anhyd.) at $\sim 500^\circ\text{C}$	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie
				See ref. under Al_2BaO_4 , p. 41.
				Same, p. 43.

(a) CdO + Cr₂O₃ heated in vacuo at 300°C for 17 hr;
 (b) ground, pelletized, heated at 1050°C for 1.5 hr.

H. Swanson
J. deGroot
H. McMurdie

See ref. under
Al₂BaO₄, p. 16.



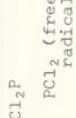
(a) crystallized from aq. soln. at 95°C;
 (b) also prep'd. from CdSO₄ or CdSO₄·8H₂O on prolonged exposure to air of RH = 50%.

obtained from Johnson, Mathey & Co., Ltd.

H. Swanson
J. deGroot
H. McMurdie



same, p. 8.



EPR spectrometry

formation of PCl₄ by sequential rxns.; evaluation of molecular bonding, relation to chlorophosphorylation via radical intermediates

G. F. Kokoszka and F. E. Brinckman, "Electron Paramagnetic Resonance Studies of Phosphorus-Containing Reactive Intermediates", Chem. Comm., 349 (1969).

T. C. Farrar

G. F. Kokoszka and F. E. Brinckman, "Electron Paramagnetic Resonance Studies of Phosphorus-Containing Reactive Intermediates", J. Am. Chem. Soc., 92, 1199 (1970).

co-pptd. CsCl
+ CsCl₂ heated
in sealed
glass tube
at 500°C

See ref. under
C₂Na₂O₄, p. 11.

C₁₃CsNi
CsNiCl₃

x-ray diffracto-
metry, petrographic
microscopy

co-pptd. NH₄Cl
+ CsCl₂ heated
in sealed
glass tube
at 500°C

See ref. under
C₂Na₂O₄, p. 5.

x-ray diffracto-
metry, petrographic
microscopy

See ref. under
C₂Na₂O₄, p. 57.

C₁₃CsPb
CsPbCl₃

dark red
hex. prisms
terminated
by bipyramids

co-crystallized
from aq.
CsCl + CuCl₂

hydrates
readily in
moist air

moderately
hygroscopic

See ref. under
C₂Na₂O₄, p. 57.

See ref. under
C₂Na₂O₄, p. 5.

C₁₃CsPb
CsSrCl₃

co-pptd. CsCl +
PbCl₂ at
~ 500°C

x-ray diffracto-
metry, petrographic
microscopy

See ref. under
C₂Na₂O₄, p. 12.

C₁₃HgHgCl
NH₄HgCl₃

fusion
SrCl₂ +
CsCl melted
at 900°C

x-ray diffracto-
metry, petrographic
microscopy

See ref. under
C₂Na₂O₄, p. 13.

C₁₃HgHgCl
NH₄HgCl₃

pptd. from
soln. of
NH₄Cl +
HgCl₂

x-ray diffracto-
metry, petrographic
microscopy

See ref. under
A₁₂BaO₄, p. 9.

(a) $\text{NiCl}_2 \cdot 2\text{H}_2\text{O} + \text{HCl}$ at $150^\circ\text{C} +$ NaCl_2	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie
(b) (a) + NH_4Cl in sealed glass tube at 300°C for 72 hr.	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie
$\text{NaHgCl}_3 \cdot 2\text{H}_2\text{O}$	moderately hygroscopic	H. Swanson J. deGroot H. McMurdie
RbNiCl_3	see Cl_{14}P	see Cl_{12}P
RbCoF_3K	pptd. from mixt. $\text{KF} +$ CoF_2 soln., washed	x-ray diffracto- metry, petrographic microscopy
$\text{Na}_2\text{Co}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$	crystallized from aq. soln. $\text{Na}_2\text{SO}_4 +$ CaSO_4 at room temp.	x-ray diffracto- metry, petrographic microscopy
CoSb_2O_6	$\text{Co}_2\text{O}_4 +$ $\text{Sb}_2\text{O}_5 +$ at 100°C for 30 min. in Au boat	x-ray diffracto- metry, petrographic microscopy
$\text{K}_2\text{Co}_2\text{K}_2\text{O}_1\text{S}_3$	$\text{K}_2\text{SO}_4 +$ Co_2O_4 melted at 600°C	x-ray diffracto- metry, petrographic microscopy

CuF₃K
KCuF₄

add CuCl₂
to excess KF
in soln. +
ppt.

H. Swanson
deGroot
H. McMurdie

See ref. under
Al₂BaO₄, p. 38.

Cu_{0.6}Sb₂
CuSb₂O₆

solid state
rxn. of CuO +
Sb₂O₅ in Ag
boat at 945°C
in vacuo for
2 hr.

H. Swanson
deGroot
H. McMurdie

See ref. under
Al₂BaO₄, p. 27.

Er₂O₃
ErVO₄

ErC₂O₄ + V₂O₅
heated at
1100°C for
30 min.

H. Swanson
deGroot
H. McMurdie

See ref. under
Al₂BaO₄, p. 29.

TMPO₃
BaPO₃F
CaPO₃F
K₂PO₃F
Na₂PO₃F

also
studied
in soln.
Na₂PO₃F +
MCl; M =
Ba, Ca, K

H. Swanson
deGroot
H. McMurdie

D. L. VanderHart,
H. S. Gutowsky, and
T. C. Farrar, "NMR
Study of BaP₃O₃:
³¹P and ¹⁹F Chemical-
Shift Anisotropies
and the Absolute Sign
of the P-P Coupling
Constant", J. Chem.
Phys., 50, 1050
(1969).

F₃FeK
KFeF₃

aq. soln.
FeCl₂ +
KF → ppt.,
washed,
heated to
400°C in
vacuo

H. Swanson
deGroot
H. McMurdie

See ref. under
C₂Na₂O₄, p. 39.

F₃Mg
KMgF₃

HF + slurry
of K₂CO₃ +
MgCO₃, evapo-
rated to
dryness, x-ray
pattern
sharpened
by heating
sample to
m.p.

See ref. under
C₂Na₂O₄, p. 42.

F_3KMn	aq. MnCl_2 + excess K_2F in soln. \rightarrow ppt.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $\text{C}_2\text{Na}_2\text{O}_4$, p. 45.
F_3MnNa	aq. MnCl_2 + excess K^+ in soln. \rightarrow ppt.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $\text{C}_2\text{Na}_2\text{O}_4$, p. 65.
NaMnF_3				
F_3NaZn	soln. ZnCl_2 + conc. soln. $\text{NaF} \rightarrow$ ppt., washed, annealed at 500°C	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under $\text{C}_2\text{Na}_2\text{O}_4$, p. 74.
NaZnF_3				
FeNbO_4	solid state rxn. of Fe_2O_3 + Nb_2O_5 in sealed Pt tube at 1000–1475°C	x-ray powder diffractometry	polymorphism R. S. Roth J. L. Waring	R. S. Roth and J. L. Waring, "Tixiolite and Other Polymeric Types of FeNbO_4 ", Am. Mineral., 49, 242 (1964).
Gd_2V	$\text{Gd}_2(\text{C}_2\text{O}_4)_3$ + V_2O_5 at 800°C for 15 min.	x-ray diffractometry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al_2BaO_4 , p. 30.
GdVO_4				
$\text{GeO}_2 \cdot 9\text{Nb}_2\text{O}_5$	$\sim 50\mu$ solid state rxn. in sealed Pt tube	x-ray powder diffractometry	stable below 1420°C, phase diagram E. M. Levin	E. M. Levin, "Phase Equilibria in the System Niobium Pentoxide-Germanium Dioxide", J. Res. NBS, 70A, 5 (1966).
$\text{Ge}_4\text{O}_9\text{Pb}$	20–40 μ solid state rxn. of BaGe_4O_9 + PbO under SECTION IV	optical and x-ray powder diffractometry	isostructural with BaGe_4O_9 C. R. Robbins SrGe_4O_9	C. R. Robbins and E. M. Levin, "Tetra-germanates of Strontium Lead and Barium of Formula Type AB_4O_9 ", J. Res. NBS, 65A, 127 (1961).
PbGe_4O_9				

$\text{Ba}(\text{C}_2\text{H}_5)_2\text{Sr}$	$\text{Ba}(\text{C}_2\text{H}_5)_2\text{Sr}$	solid state rxn. of SrO + GeO_2 in sealed Pt tube at 1200°C .	species) and x-ray powder diffractometry	isostructural $\text{Cs}_x\text{K}_y\text{GeO}_z$ with K_2GeO_3 , PbGe_2O_5 .	$\text{Cs}_x\text{K}_y\text{GeO}_z$ and PbGe_2O_5 .
$\text{H}_3\text{MnO}_7\text{P}_6$	$\text{M} = \text{Ba}, \text{Pb}, \text{Sr}$	$\text{Ba}_{10}\text{P}_6\text{O}_{24}(\text{OH})_2$ $\text{Pb}_{10}\text{P}_6\text{O}_{24}(\text{OH})_2$ $\text{Sr}_{10}\text{P}_6\text{O}_{24}(\text{OH})_2$	solid state rxn. of BaO , PbO , SrO + P_2O_5 in air at elevated temp.	x-ray diffraction; infrared, broad-line NMR spectrometry; gravimetric anal.	illustrate nature of non-stoichiometric phases
H_4PI		$\text{PH}_3 + \text{HI} \rightarrow \text{PH}_4\text{I}$	infrared, NMR ($^1\text{H}, ^3\text{P}$) spectroscopy, vapor pressure	phase transitions and molecular motions in solids; toxic, air/moisture sensitive	T. C. Farrar and J. C. Roth, "High Temperature Dehydroxylation of Apatitic Phosphates", <i>J. Res. NBS</i> , 72A , 783 (1968).
H_4PX	$X = \text{Br}, \text{Cl}, \text{I}$	$\text{PH}_3 + \text{HX} \rightarrow \text{PH}_4\text{X}$	NMR ($^1\text{H}, ^3\text{P}$), laser Raman spectrometry; vapor pressure, neutron inelastic scattering	model compounds, phase transitions and motions in solids	T. C. Farrar and J. C. Roth, "Proton Magnetic Resonance and Hindered Rotation in Phosphonium Halides", <i>J. Chem. Phys.</i> , 49 , 4403 (1968).

$K_2Ni_2O_1 \cdot S$
 $K_2Ni_2O_1 \cdot O_{12}S_3$

$K_2SO_4 + Ni_2O_4$,
heated at
750°C, cooled
slowly, ground,
annealed at
550°C for
30 min.

$H.$ Swanson
J. deGroot
H. McMurdie

x-ray diffracto-
metry, petrographic
microscopy

$K_2O_4S_3Zn_2$
 $K_2Zn_2(SO_4)_3$

$K_2SO_4 + ZnSO_4$,
melted, ground,
remelted

$H.$ Swanson
J. deGroot
H. McMurdie

$K_3Na_0.8S_2$
 $K_3Na(SO_4)_2$

glaserrite
stoichiometric K_2SO_4
+ Na_2SO_4 ,
melted,
annealed at
700°C for
72 hr.

$H.$ Swanson
J. deGroot
H. McMurdie

$K_2SO_4 \cdot H_2O$

x-ray diffracto-
metry, petrographic
microscopy

$H.$ Swanson
J. deGroot
H. McMurdie

$LiNaO_4S$
 $LiNaSO_4$

equimolar
 Li_2SO_4 +
 Na_2SO_4 ,
melted,
annealed
at 500°C
overnight

$H.$ Swanson
J. deGroot
H. McMurdie

$LiNbO_3$

x-ray diffracto-
metry, petrographic
microscopy

$H.$ Swanson
J. deGroot
H. McMurdie

Li_2O_4S
 Li_2SO_4

obtained
from CIBA,
Rare Metals
Div., Summit,
N.J.; re-
crystallized
by W. S.
Broer,
pulled from
melt, anneal-
ed under O_2
at 1100°C for
10 hr.

$H.$ Swanson
J. deGroot
H. McMurdie

x-ray diffracto-
metry, petrographic
microscopy

$H.$ Swanson
J. deGroot
H. McMurdie

$Li_2O_4 \cdot H_2O$

heated at
600°C for
24 hr.

$H.$ Swanson
J. deGroot
H. McMurdie

x-ray diffracto-
metry, petrographic
microscopy

$H.$ Swanson
J. deGroot
H. McMurdie

$Li_2O_4 \cdot H_2O$

heated at
600°C for
24 hr.

$H.$ Swanson
J. deGroot
H. McMurdie

Lu ₂ O ₄ V	soln. Na ₃ VO ₄ + Lu ₂ (SO ₄) ₃ + ppt., annealed at 1300°C for 15 hr.	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie	See ref. under Al ₂ BaO ₄ , p. 37.
Na ₂ NiO ₈ S ₂ ·4H ₂ O	Na ₂ Ni(SO ₄) ₂ · 4H ₂ O	crytallized from aq. soln. of Na ₂ SO ₄ + NiSO ₄ at room temp.	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie
Na ₂ O ₈ S ₂ Zn·4H ₂ O	Na ₂ Zn(SO ₄) ₂ · 4H ₂ O	crytallized from soln. of Na ₂ SO ₄ + ZnSO ₄ at room temp.	x-ray diffracto- metry, petrographic microscopy	H. Swanson J. deGroot H. McMurdie
NbO ₄ Sb		solid state rxn. of Sb ₂ O ₃ + Nb ₂ O ₅ in sealed Pt tube at 1000- 1100°C	see BiNbO ₄	R. S. Roth J. L. Waring
SiNbO ₄				See ref. under BiNbO ₄ .
"NbO ₅ P"	"NbPO ₅ " 2Nb ₂ ·P ₂ O ₅	"high-low" ~ 50 _u	solid state rxn. of Nb ₂ O ₅ +P ₂ O ₅ in sealed Pt tube	x-ray powder diffractometry
Nb ₂ O ₆ Ti	TiNb ₂ ·O ₆	10-25 _u	solid state rxn. of TiO ₂ + Nb ₂ O ₅ at 1450°C	x-ray powder and single crystal diffractometry; crystal structure
				systematics of "non- stoichiometry"
				R. S. Roth

$\text{Ru}_{1-x}\text{SbTa}_x$	powder	$(1-x)\text{O}_2\text{Th} + x\text{CaO}$ $0 < x \leq 0.10$	porous bar	isostatically cold pressed and sintered 1 hr. at 1800°C	x-ray diffractometry, phase equilibrium spectroscopic analysis	illustrates behavior of Ru by heat treatment in air	C. L. McDaniel S. J. Schneider	See ref. under IrO_3 .
IrO_2					character of point defect; electrical and mechanical relaxation	dissociates at 1045°C in air to $\text{Ru} + \text{O}_2$; see also IrO_2	J. Wachtman, Jr.	"J. B. Wachtman, Jr., and W. C. Corwin, "Internal Friction in ThO_2 Containing CaO ", <i>Phys. Rev.</i> , <u>131</u> , 517 (1963)."
$\text{Pr}_2(\text{C}_2\text{O}_4)_3 + \text{V}_2\text{O}_5$ heated at 850°C for 30 min.	porous bar	$(1-x)\text{O}_2\text{Zr} + x\text{CaO}$ $0.04 \leq x \leq 0.20$	porosity, grain size	porosity, grain size	x-ray diffraction, petrographic microscopy	spans range of stability of cubic solid solution and includes 2-phase region below $x=10$ as test of changing in character of point defects.	J. Wachtman, Jr.	"J. B. Wachtman, Jr., and W. C. Corwin, "Internal Friction in ZrO_2 Containing CaO ", <i>J. Res. NBS</i> , <u>69A</u> , 457 (1965)."
PrVO_4							E. Swanson deGroot H. McMurdie	R. S. Roth J. L. Warling
SbTaO_4								See ref. under Al_2BaO_4 . See ref. under BiNbO_4 .
SbVO_4								R. S. Roth J. L. Warling

O₄ SmV
SmVO₄

Sm₂(C₂O₄)₃ +
V₂O₅ heated
at 850°C for
45 min.

H. Swanson
deGroot
H. McMurdie

See ref. under
Al₂BaO₄, p. 47.

O₄TbV
TbVO₄

Tb₂(C₂O₄)₃ +
V₂O₅ heated
at 1000°C
for 1 hr.

x-ray diffracto-
metry, petrographic
microscopy

See ref. under
Al₂BaO₄, p. 56.

O₄TmV
TmVO₄

Tm₂(C₂O₄)₃ +
V₂O₅ heated
at 1400°C
for 1 hr.

x-ray diffracto-
metry, petrographic
microscopy

See ref. under
Al₂BaO₄, p. 57.

O₄VYb
YbVO₄

Yb₂(C₂O₄)₃ +
V₂O₅ heated
at 1400°C
for 1 hr.

x-ray diffracto-
metry, petrographic
microscopy

See ref. under
Al₂BaO₄, p. 58.

59

Pt

special
prep. for
Office of
Standard
Reference
Materials,
proposed SRM
747

Langmuir detm.
of vapor pressure
as function of
temp.; detm. of
heat of sublimation

See ref. under Ag.

SRM # 747
SRM # 680

vapor pressure
Langmuir method

See above ref.

Re

rod,
.25 cm.
dia. x
1.5 cm.

evaluated as
container for
high-temp.
studies (up
to 2000K);
vapor pressure
standard

E. R. Plante
See above ref.

Re

commercial,
zone refined

vapor pressure

E. R. Plante
See above ref.

E. R. Plante
R. Szwarc, "Vapor
Pressure and Heat of
Sublimation of
Rhenium," J. Res.
NBS, 70A, 175
(1966).

$\text{V}_2\text{O}_5 \cdot 4\text{Y}_2\text{O}_3$	$\sim 50\%$	solid state rxn. in sealed Pt tube	x-ray powder diffractometry	phosphors	E. M. Levin	E. M. Levin, "System $\text{Y}_2\text{O}_3 \cdot \text{V}_2\text{O}_5$ ", J. Am. Ceram. Soc., 50, 381 (1967).
$\text{V}_2\text{O}_5 \cdot 5\text{Y}_2\text{O}_3$						
$5\text{Y}_2\text{O}_3 \cdot \text{V}_2\text{O}_5$						

W	high-purity rod, 1/4 in. dia. x 2-1/2 in.	special prep. for Office of Standard Reference Materials, proposed SRM # 749	see Pt	see Pt	R. C. Paule	See ref. under Pt.

$\text{poly}-$ stalline	commercial rod	vapor pressure, Langmuir method	vapor pressure standard; container for high-temp. studies	E. R. Planté	R. Szwarc, E. R. Planté, and J. J. Diamond, "Vapor Pressure and Heat of Sublimation of Tungsten", J. Res. NBS, 69A, 417 (1965).
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COMPOSITION	NATURE OF PREPARATION	METHOD OF CHARACTERIZATION	COMMENTS	
$\text{Al}_2\text{O}_3\text{-WO}_3$ entire system	polycrystalline line, 1-25 μ	solid state rxn. of components in sealed Pt tubes	phase equilibria; x-ray powder and single crystal diffraction studies	J. L. Waring J. L. Waring and R. S. Roth, "Phase Equilibrium in the System Aluminum Oxide-Tungsten Oxide", <i>J. Am. Ceram. Soc.</i> , <u>48</u> , 493 (1965).
$\text{As}_2\text{O}_5\text{-Nb}_2\text{O}_5$	polycrystalline line, 1-25 μ	solid state rxn. in sealed Pt tube	phase equilibria; x-ray diffraction powder and single crystal structure	J. L. Waring R. S. Roth
$\text{As}_2\text{O}_5\text{-Nb}_2\text{O}_5$ partial system	polycrystalline line, 1-25 μ	solid state rxns. of mixtures of M_2O_3 + B_2O_3 in sealed Pt tubes	phase equilibria; x-ray diffractionometry; high-temp. x-ray phase equilibria	J. L. Waring and R. S. Roth R. S. Roth and A. D. Wadsley, "The Crystal Structure of $\text{Pb}_{0.5}\text{Nb}_{0.5}$ apparently isostructural with Ta_2O_5 ; see also: $\text{As}_{0.5}\text{-9Ta}_2\text{O}_5$, $\text{Ge}_{0.2}\text{-9Nb}_2\text{O}_5$, $9\text{Nb}_{2.0}\text{-5P}_2\text{O}_5$, $9\text{Nb}_{2.0}\text{-5V}_2\text{O}_5$, $2\text{Nb}_{2.0}\text{-Ta}_{2.0}\text{O}_5$, $\text{P}_{2.0}\text{-9Ta}_{2.0}\text{O}_5$, $9\text{Ta}_{2.0}\text{-5V}_{2.0}\text{O}_5$
$\text{As}_2\text{O}_5\text{-9Ta}_2\text{O}_5$	partial system, see comments and ref. under $\text{As}_2\text{O}_5\text{-9Nb}_2\text{O}_5$	solid state rxns. of mixtures of M_2O_3 + B_2O_3 in sealed Pt tubes	x-ray powder diffractometry; high-temp. x-ray phase equilibria	R. S. Roth, J. L. Waring, and E. M. Levin, "Polymerism of ABO_3 -Type Rare Earth Borate Solid Solutions", <i>Rare Earth Research II</i> , Gordon & Breach, N.Y., (1964), p. 153.
BO_3M MBO_3	solid soins. of rare-earth borates, $\text{M} = \text{Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Sm, Tm, Y, Yb}$; polycrystalline line	solid state rxns. of mixtures of M_2O_3 + B_2O_3 in sealed Pt tubes	most specimens exhibit polymorphic inversions	R. S. Roth, J. L. Waring, and E. M. Levin

LaFeO_3 -n BaO- "iron oxide"	solid state rxn. in Au tubes under O_2 ratio; poly- crystalline, 1-25 μ	phase equilibria; x-ray diffractometry of 1:1 cation tubes	T. Negas and R. S. Roth, "Synthesis of Barium Ferrates in Oxygen", J. Res. NBS, 73A, (1969).
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$\text{BaO-Nb}_2\text{O}_5$ entire system	polycrystal- line	solid state rxns. of com- ponents in sealed Pt tubes	R. S. Roth J. L. Waring "Phase Equi- librium Relations in the Binary System Barium Oxide-Niobium Pentoxide", J. Res. NBS, 65A, 337 (1961).
Ba-Pt entire system	powders	solid state rxn. conducted in air in Pt and Au con- tainers	x-ray diffracto- metry; phase equilibria illustrates behavior of container material

$\text{BaSi}_2\text{O}_5-\text{Ba}_2\text{Si}_3\text{O}_8$ entire system	polycrystal- line	solid state rxn.	R. S. Roth E. M. Levin "Phase Equi- libria in the System Barium Disilicate- Dibarium Trisilicate", Am. Mineral., 62, 193 (1969).
BaSi ₂ O ₅ -Ba ₂ Si ₃ O ₈ entire system	polycrystal- line	solid state rxns. in sealed Pt tubes	x-ray powder diffractometry; phase equilibria; single crystal x-ray diffractometry

$\text{Bi}_2\text{O}_3-\text{Nb}_2\text{O}_5$ entire system	polycrystal- line	solid state rxns. in sealed Pt tubes	R. S. Roth J. L. Waring "Phase Equi- librium Relations in the Binary System Bismuth Sesquioxide- Niobium Pentoxide", J. Res. NBS, 66A, 451 (1962).
Ba-Pt entire system	powders	solid state rxn. conducted in air in Pt and Au con- tainers	x-ray diffracto- metry; phase equilibria illustrates behavior of container material

Cr_2O_3 - IrO_2 entire system	powders	solid state rxns. in Pt, Vycor; Ir containers, conducted in air	x-ray diffractometry; phase equilibria	illustrates behavior of container materials	C. L. McDaniel and S. J. Schneider
CrO_n - SrO "chromium oxide" - SrO entire system	polycrystal-line, 1-25 μ	solid state rxns. in air and O_2	x-ray diffractometry; gravimetric anal.; phase equilibria	illustrates T. Negas and R. S. Roth redox behavior R. S. Roth	T. Negas and R. S. Roth, "The System SrO -"chromium oxide" in Air and Oxygen", J. Res. NBS, 73A (1969).
Dy_2O_3 - TiO_2	partial system	see comments and ref. under Gd_2O_3 - TiO_2			
Er_2O_3 - 2TiO_2	partial system	see comments and ref. under Gd_2O_3 - TiO_2			
Eu_2O_3 - TiO_2 Eu_2O_3 - 2TiO_2	partial system	see comments and ref. under Gd_2O_3 - TiO_2			
Gd_2O_3 - TiO_2 entire system	polycrystal-line, 1-25 μ	solid state rxn. in unsealed Pt tubes	x-ray diffractometry; polarizing microscopy; phase equilibria	illustrates nature of phases formed and relationship to analogous systems; see also:	J. L. Waring and S. J. Schneider, "Phase Equilibrium Relationships in the System Gd_2O_3 - TiO_2 ", J. Res. NBS, 79A, 255 (1965).
GeO_2 - $9\text{Nb}_2\text{O}_5$	partial system	see comments and refs. under As_2O_5 - $9\text{Nb}_2\text{O}_5$; also see GeO_2 - $9\text{Nb}_2\text{O}_5$ in SECTION V			
Ho_2O_3 - 2TiO_2	partial system	see comments and ref. under Gd_2O_3 - TiO_2			

TiO ₂ -SnO ₂	powders	solid state rxns. in Pt, Vycor, and Ir containers, conducted in air	x-ray diffractometry, phase equilibria	illustrates behavior of container materials	C. L. McDaniel and S. J. Schneider, "Phase Relations in the Systems TiO ₂ -IrO ₂ and SnO ₂ -IrO ₂ in Air", J. Res. NBS, <u>71A</u> , 119 (1967).
Ln ₂ O ₃ -PdO	powders	solid state rxns. in Pt, Vycor containers	x-ray diffractometry; phase equilibria	all intermediate phases dissociate; illustrates behavior of container materials	C. L. McDaniel and S. J. Schneider, "Phase Relations between Palladium Oxide and the Rare-Earth Sesquioxides in Air", J. Res. NBS, <u>72A</u> , 27 (1968).
entire system					
entire system					
La ₂ O ₃ -2TiO ₂		partial system, see comments and ref. under Gd ₂ O ₃ -TiO ₂			
9Nb ₂ O ₅ -P ₂ O ₅		partial system, see comments and refs. under As ₂ O ₅ -9Nb ₂ O ₅ ; also see "Nb _{0.5} P" in SECTION V			
entire system					
2Nb ₂ O ₅ -Ta ₂ O ₅		partial system, see comments and refs. under As ₂ O ₅ -9Nb ₂ O ₅			
Nb ₂ O ₅ -V ₂ O ₅		polycrystalline, 1-25 μ	x-ray powder diffraction; phase equilibria	illustrates nature of multi-phase formation and non-stoichiometric phases; see also 9Nb ₂ O ₅ -V ₂ O ₅	J. L. Waring and R. S. Roth, "Phase Equilibrium in the System Vanadium Oxide-Niobium Oxide", J. Res. NBS, <u>69A</u> , 119 (1964).
entire system		sealed Pt tubes			
9Nb ₂ O ₅ -V ₂ O ₅		partial system, see comments and refs. under As ₂ O ₅ -9Nb ₂ O ₅ ; also see Nb ₂ O ₅ -V ₂ O ₅			
P ₂ O ₅ -9Ta ₂ O ₅		partial system, see comments and refs. under As ₂ O ₅ -9Nb ₂ O ₅			
Sm ₂ O ₃ -TiO ₂		partial system, see comments and ref. under Gd ₂ O ₃ -TiO ₂			

$Ta_2O_5-TiO_2$
entire system

polycrystal-line
solid state rxns.
sealed Pt tubes

x-ray powder diffractometry; high-temp. x-ray powder diffractometry; phase equilibria illustrates various stable and metastable phases formed in the system

$9Ta_2O_5-V_2O_5$

partial system, see comments and refs. under $As_2O_5-9Nb_2O_5$

$2TiO_2-Tm_2O_3$

partial system, see comments and ref. under $Gd_2O_3-TiO_2$

J. L. Waring and R. S. Roth, "Effects of Oxide Additions on the Polymorphism of Tantalum Pentoxide (System $Ta_2O_5-TiO_2$)", J. Res. NBS, 72A, 175 (1967).

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